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THE METAMICT STATE*

A. PABST, *University of California, Berkeley 4, California.*

ABSTRACT

The development of investigation of the metamict state is considered. Observations of the changes induced by heating metamict thorite are reported.

INTRODUCTION

The word "metamict" has been widely used in recent years. It appears several times in the first volume of the 7th edition of "Dana's System" and yet one will look in vain for a definition of it in dictionaries or encyclopedias and in most mineralogical textbooks.[1] Hence it may be of interest to refer briefly to the origin of this word.

"Metamict" was defined by Broegger in a publication not readily accessible to or generally known by mineralogists. The definition appears near the end of a thousand word article on "amorf" in the first edition of Salmonsens Konversationslexikon published in Danish in Copenhagen in 1893.[2] After pointing out the characteristic differences between crystalline and amorphous solid substances, Broegger referred to the fact that Breithaupt had long ago divided the latter into *porodine* amorphous substances, formed by slow hardening of gelatinous masses, for example opal, and *hyaline* amorphous substances, formed by rapid cooling from a molten state, for example obsidian. In addition to these two principal groups of amorphous substances Broegger recognized a third. Certain originally crystalline substances, he said, have in the course of time assumed the properties of amorphous substances. While retaining their original crystalline form they have become lighter in specific gravity, assumed conchoidal fracture, have become optically isotropic and so on. He enumerated some of these materials, such as gadolinite, euxenite, fergusonite, thorite, zircon and others, many containing the rare earth elements and thorium or uranium. Some, for instance thorite, he said, are known only in the amorphous state.

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* Address of the retiring President of the Mineralogical Society of America given at the thirty-second meeting of the Society in Detroit, Michigan, Nov. 8, 1951.

[1] An exception is Klockmann's *Lehrbuch der Mineralogie* by Paul Ramdohr, 13th ed., 1948, where the word is defined by implication on page 227.

[2] Incidentally we may be reminded that another mineralogical matter of far greater importance was first developed in an encyclopedia. The first derivation of the 32 symmetry classes was given by J. F. C. Hessel in a 200 page article on "Krystall" in the fifth volume of Gehler's *Physikalisches Wörterbuch*, 1830, and it may well be that this obscure manner of publishing delayed the general recognition of the importance of the 32 classes until the work of Gadolin nearly 40 years later.

Broegger's definition follows:

> "For this third class of amorphous substances there is herewith proposed the name "metamict (from *μεταμιγνυμ*, mix otherwise, that is by a molecular rearrangement to another molecular structure than the original crystalline) amorphous substances. The reason for the amorphous rearrangement of the molecules might perhaps be sought in the lesser stability which so complicated a crystal molecule as that of these minerals must have in the presence of outside influences."

STUDY OF THE METAMICT STATE

Broegger (1890) had been interested in isotropized materials for several years and had listed a large number in his monograph on the nepheline syenite pegmatites. Petersson (1890) did his work on gadolinite in part in Broegger's laboratory. He showed that isotropic and anisotropic gadolinite have essentially the same composition and that isotropic gadolinite could be made anisotropic at a certain temperature at which it shows the phenomenon of "glowing," this being a sudden incandescence which passes through the mineral when it is warmed to a moderate temperature and must be attributed to evolution of heat within the mineral itself.

This property of "glowing" which characterizes certain metamict substances was first reported by Berzelius (1815) who stated that his attention was called to it by Wollaston. Berzelius observed that gadolinites though differing in their behavior before the blowpipe all show the property of "glowing." He wrote:

> "If a piece of gadolinite is slowly heated before the blowpipe to incipient incandescence so that the whole piece is warmed uniformly as far as possible, at a certain temperature it suddenly comes to ignition as though it took fire and this ignition will spread over the whole mass the more rapidly the more uniformly the piece has been heated."

The word "pyrognomic," meaning "readily becoming incandescent on heating," was coined by Scherrer (1840) to designate materials showing this behavior and it is still to be found in modern dictionaries although it is scarcely used by present day mineralogists.

Pyrognomic behavior was soon found in various minerals. For instance, Shepard (1851) recorded that a material he called rutherfordite, later shown to be fergusonite, "when heated in a glass tube by means of blowpipe—cracks to pieces, glows as if on fire, emits much moisture, and turns yellow."

In the course of time other changes produced by heat in the minerals which Broegger later classified as metamict were noted or investigated by mineralogists. Des Cloiseaux and Damour (1860) reported that optically isotropic gadolinite becomes birefringent after heating and made similar observations on a number of other substances. Damour (1864) recorded the increases in specific gravity produced by heating of zircons.

After Broegger various writers have tabulated the characteristics of metamict materials. In doing this they have usually proceeded from an acquaintance with only a few metamict materials and only a partial knowledge of the findings of other investigators. This has led to faulty and over-simplified summaries of the properties of metamict substances and especially to the attribution of the properties of particular species or even of particular specimens to metamict substances generally. Commenting on his recent summary Faessler (1942) says that not all of the properties he lists belong to all metamict materials but does not indicate just how this qualification is to be applied to the several items on his list. The following annotated list of the properties of metamict minerals has been compiled from many sources. It is valid only with the annotations.

Properties of Metamict Minerals

1. Optically isotropic.	Many metamict substances are heterogeneous being partly isotropic and partly anisotropic.
2. Pyrognomic, i.e., readily becoming incandescent on heating.	In this regard there is great variation, Liebisch (1910), with no glowing observable in some cases. Even strongly pyrognomic minerals like gadolinite can be annealed below the temperature of glowing with complete loss of the pyrognomic quality, Faessler (1942). Prior (1894) had previously observed the restitution of birefringence in fergusonite by heating with no pyrognomic effect.
3. Lacking in cleavage, fracture conchoidal.	Some, also, are particularly brittle.
4. Density increased by heating.	This is commonly the case but the change is slight in some species and misleading reports have been given for a few on account of difficulties of accurate measurement, Scherrer (1840) and Rose (1843).
5. Crystalline structure reconstituted by heating.	This conclusion was drawn from the optical changes noted by Des Cloiseaux and later workers. In some cases it is suggested that a single crystal is reformed (e.g. Ramdohr 1948). Reconstitution may be a very complex process and even where a single species results may not yield single crystals.
6. Resistance to attack by acid increased by heating.	This curious effect of heating, just the opposite of heating to fusion in many instances, was found by Rose (1841), also noted by Petersson (1890) and investigated by Bauer (1939).

7. Contain U or Th.	The content of U or Th may be low, e.g. 0.41% ThO_2 in gadolinite from Ytterby. The presence of rare earths and related elements has been emphasized by some workers.
8. Some minerals are known in both the crystalline and metamict states.	In these cases little if any chemical difference can be found. There is evidence of hydration attending isotropization but no direct or general correlation has been established. Des Cloiseaux and Damour (1860) found allanites (orthites) might be anhydrous and either isotropic or birefringent or hydrated and either isotropic or birefringent.
9. *X*-ray amorphous.	Vegard (1916) first reported the absence of *x*-ray diffraction in thorite. This has since been noted for many other metamict minerals. Some show traces of *x*-ray diffraction though optically isotropic.

Numerous causes have been assigned to metamictization or isotropization. Some early workers considered that the isotropic and birefringent forms correspond to dimorphs, Des Cloiseaux and Damour (1860). In recording the specific gravity increases on heating zircons, Damour (1864) referred to "l'allotropie des zircons." A little later the variation of density in zircons was even considered to be possibly due to the presence "instead of zirconium" of "another closely allied element," Stevanović (1902), Spencer (1904). Others have considered that the isotropic paramorphs arise "as a result of hydration" and this is expressly stated by Lacroix (1922–23) in the discussion of several metamict minerals.

Broegger attributed metamictization to unspecified "outside influences." He considered that the minerals which become metamict are especially susceptible to these influences because of the lesser stability of their "complicated crystal molecule." The notion of lesser stability has been elaborated upon by several later workers though it is no longer thought of as necessarily connected with complex composition. There are many materials that are of complex composition but not known to be metamict. On the other hand some metamict substances have a relatively simple composition.

The suggestion that the isotropization of metamict minerals might have been effected by radioactive emanations was first made by Hamberg (1914). He seems to have been led to this after considering the effects of alpha radiation in pleochroic halos. Even before the discovery of radioactivity, Michel-Lévy (1889) had observed that birefringence as well as color is affected in these halos. The reduction or loss of birefringence was the optical change most frequently noted by early students of metamict minerals, measurements of refractive indices being rarely made in those days before the introduction of the immersion method.

Recognition that alpha radiation might be involved in the process of metamictization did not solve the problem at all. It remained to explain why alpha bombardment is effective in some cases and not in others.

The condition for metamictization given by Broegger (1893), that minerals so affected must have a special predisposition thereto, was restated in phrases appropriate to a later era by Goldschmidt (1924). He enumerated three conditions required for metamictization: *A*. The original structure must be only weakly ionic and possibly susceptible to hydrolysis. *B*. The structure must contain one or more kinds of ions that are readily susceptible to changes in the state of ionization. *C*. In many cases it may additionally be necessary that the crystal is subjected to relatively *strong* radiation, either from radioactive material within the crystal itself or from outside sources. No one of these conditions is alone sufficient for isotropization. It may be noted that materials of strongly ionic binding are not known in the metamict state. This state is characteristic of isodesmic multiple oxides as recently pointed out by Holland and Kulp (1950). That natural irradiation, however strong, will not necessarily render a mineral metamict is shown by the existence of fully crystalline thorianite and pitchblende.

If the complicated composition of metamict minerals renders them unstable, as initially proposed by Broegger, one might suppose that this is due to the fact that extended isomorphous substitutions have a limited range of stability. Upon change of temperature and pressure from the conditions under which they formed these substances may become unstable. It was pointed out by Machatschki (1929) that this, however, leads to exsolution in many well-known cases. A few years later Machatschki (1941) suggested that for zircon the instability making it susceptible to metamictization might be sought in the eight coordination of its zirconium. This element has an ionic radius close to the limit of the ranges associated with eight- or six-fold coordination and is found in six coordination in many materials.

In a recent review of the "nature and cause of the metamict state" Hutton (1950) has emphasized the suggestion of Tomkieff (1946) that changes in ionic radius attending oxidation of uranium will explain the metamictization of uraninite. This is considered to follow from Ellsworth's (1925) statement that "all minerals containing UO_2 automatically oxidize themselves at a rate depending on the rate of uranium and thorium disintegration." To me it seems that these authors have not considered the processes involved after a state of equilibrium has been reached, that is, when uranium is in equilibrium with its series of decomposition products and lead atoms are appearing at the same rate at which uranium atoms are disintegrating. Conybeare and Ferguson (1950) and Brooker and Nuffield (1951) have recently studied the correlation between the state of oxidation of uranium in pitchblendes and their degree of metamictness.

The characteristic behavior of pyrognomic minerals may be considered to be caused by the liberation of heat as they change rapidly from the higher energy metamict state to a crystalline state, the energy released having been acquired in the process of isotropization. According to current views, the source of this energy is to be sought in the radioactive decay of constituents of the minerals themselves. Radioactive materials outside the affected minerals could hardly be expected to have the observed effect since the range of natural alpha particles in the materials concerned is of the order of 0.02 mm. (Stackelberg and Rottenbach, 1940).

The evolution of heat associated with the "glowing" of pyrognomic minerals was first demonstrated by Rose (1843). The first careful measurements were made by Liebisch (1910), who gave heating curves for a number of pyrognomic minerals and found that a rough correlation could be made between the generation of heat indicated by these curves and the visibility of the luminescence. He found, as might have been expected, that gadolinite, the first known pyrognomic mineral and the one showing the liveliest glow, gives the most pronounced break in the heating curve. The study of the heat effect was made quantitative by Faessler (1942) who managed to measure the heat of transformation by direct calorimetry on gadolinite from Ytterby which had been the subject of study by so many others. The heat of transformation was found to be of the same order of magnitude as the heat of crystallization of comparable materials. Incidentally Faessler carried out more precise and extensive measurements of the specific heats of reconstituted and of metamict gadolinite than had previously been made, and found that the relations of these specific heats are similar to those of crystalline silicates and their glassy equivalents.

Recently the heat effects shown by metamict minerals have been recorded by differential thermal curves (Kerr and Holland, 1951) and the use of these curves for purposes of identification has been suggested.

There has been some speculation as to the reasons for variations in degree of metamictness of different samples of the same mineral. Hutton (1950) has asked whether certain thorites might be not "old enough for the transformation to a metamict state to have taken place." Mügge (1922) noted that artificial U, Th and Ra salts show no metamictness, nor do such minerals in young rocks show it, e.g., it is not observed in zircons or allanites of young volcanics. A study of zircons from granitic rocks of various ages by Morgan and Auer (1941) showed that the degree of metamictness as indicated by change in optical properties could be correlated with radioactivity of the samples but this bears no simple relation to age.

The degree of metamictness has been estimated by Chudoba and

Lange (1949) from changes in density. Faessler (1942) considered time-temperature curves suitable for making such an estimate though his own observations do not seem to me to be in harmony with this opinion. Recently Holland and Kulp (1950) have suggested that an estimation of the degree of metamictness from differential thermal analysis might be used for age determination. They say:

> "It seems plausible to assume with previous authors that these minerals were originally crystalline, and that destruction of the crystal lattice has taken place since their formation through alpha bombardment from the uranium and thorium in the lattice.
>
> "If this is the true mechanism, it follows that the amount of destruction of the lattice is affected by (1) the inherent stability of the given mineral structure, (2) the total alpha activity of the mineral, and (3) the time elapsed since the formation of the mineral. If the first two variables can be specified and if the degree of destruction of the lattice can be quantitatively measured, then the age is uniquely determined.
>
> "The amount of disorder created by alpha bombardment of the lattice can be measured by means of thermal analysis, since the area under the exothermic peak on the thermal record will be proportional to the amount of heat generated in the specimen upon returning to the crystalline state."

Little is known of the efficiency of alpha bombardment in the destruction of a crystal. The matter has recently been touched upon by Hurley (1951). He says:

> "The exact nature of the structural disturbance caused by heavy ionizing particles is not known. It is well known . . . , that *x*-ray diffraction patterns are obliterated under heavy dosages of alpha particles. If the process in solids is at all similar to that in gases, an alpha particle will lose energy chiefly by interaction with electrons and not by nuclear reactions or scattering. According to classical theory, each alpha particle would initiate in the order of 10^5 secondary electrons of mean energy 100-200 ev. before coming to rest. This would leave a cylindrical volume along the track intensely irradiated by these electrons and those which they in turn displaced. In a non-conductor such as a silicate mineral the electrons might remain out of place, and the presence of the extra electrons as well as the disrupted bonds might displace the atoms or at least leave them in a weakly bound condition.

A calculation of the time required to make a given mineral metamict is possible if we make certain simplifying assumptions. Let us assume:

A. That the entire energy from radioactive decay is spent in disruption of the crystal structure.

B. That the "heat of transformation" of the metamict to the crystalline state is a correct measure of the energy differences of the two states, that is, of the energy which was required for disruption of the crystal structure.

We have the necessary data to make the calculation for gadolinite from Ytterby.

1. The heat of transformation of the gadolinite. This was determined by Faessler (1942) to be 89.1 calories per gram.
2. The concentration of thorium in the gadolinite. At 0.41% ThO_2 found by Petersson (1890) this is about 0.004 gram per gram.

3. The energy provided by thorium in equilibrium with its decay products. According to Evans and Goodman (1941) this is 0.203 calorie per gram per year.

Dividing 1 by the product of 2 and 3 we find that under the assumed conditions Ytterby gadolinite would become entirely metamict in about 110,000 years. If one makes a corresponding calculation for an hypothetical mineral having similar thermal properties to gadolinite and containing one per cent of uranium one finds that only 12,000 years would be required for complete metamictization.

Assumption *A* made above is probably not justified. No doubt only a part of the radioactive energy is effective in disruption of the crystal structure and this process takes much longer than the time calculated. On this point Hurley (1951) says:

> "Differential thermographic measurements are being made to determine the amount of heat given off as zircon becomes ordered again at elevated temperature, as a part of a study of the proportion of alpha particle energy that goes into permanent disturbance of the structure."

It is, however, not likely that all metamict materials will lend themselves so well to the determination of the heat of transformation as the markedly pyrognomic gadolinite. The changes produced by heating metamict minerals may be very slow and in some cases do not lead to the simple reconstitution of the original structure.

Until the discovery of *x*-ray diffraction the best method for studying the changes effected in a metamict material by heat was observation under the polarizing microscope. Several investigators reported the reappearance of double refraction and some observed what was interpreted to be the reconstitution of a single crystalline edifice in proper orientation with the external form. As Mügge (1922) said "Es mutet das den Kristallographen an wie die Auferweckung eines Toten." Mügge made very careful observations on the progressive changes in double refraction and showed that these indicated that intermediate stages may consist of a web- or sponge-like crystal whose meshes are filled with amorphous substances, the aggregate effect being that of a single crystal without fully restored birefringence.

X-Ray Studies

Vegard (1916) was the first to note the absence of *x*-ray diffraction in a metamict mineral (thorite). In later years many investigators have studied the reconstitution of metamict minerals by means of *x*-rays. Commonly powder methods only have been used and thus the possibility of recognizing fully what goes on within an apparent single crystal has been missed. Even such limited observations did yield interesting results. Goldschmidt (1924) found that fergusonite is reconstituted by heating whereas thorite is not reconstituted by "brief heating."

The use of single crystal patterns by v. Stackelberg and Chudoba (1937) and v. Stackelberg and Rottenbach (1940) showed that single crystals are indeed produced by the heating of some zircons that are partly metamict, but they also found that a fully amorphous zircon after suitable heat treatment may yield a ZrO_2 diffraction pattern and on further heating a zircon fiber pattern with a large measure of disorientation. The Ytterby gadolinite also yields but a fiber pattern after heating 10 hours at 950° C. according to Chudoba and Lange (1949) and the orientation is worsened by heating 16 hours at 1000° C. They further reported the complete reconstitution of both samarskite and euxenite at well below the pyrognomic temperature. Arnott (1950) speaking of metamict material which has presumably regained its original structure after heating says "the resultant material, however, is cryptocrystalline." It seems probable that single crystals can be reconstituted only when there are remnants of the original structure and even then only in certain cases.

Observations on Thorium Silicate

Not long ago Professor Hutton discovered a monoclinic form of natural thorium silicate which I had the pleasure of naming huttonite. It is found in minute clear crystal fragments not in the least metamict, (Pabst and Hutton, 1951). Previously thorium silicate had been known naturally only as the tetragonal mineral thorite and only in the metamict state. Shortly after the discovery of natural monoclinic thorium silicate Hutton kindly gave me some minute single crystals of thorite containing 11.5% UO_2 and designated uranothorite. These proved to be not metamict, showing only a slight streaking in the single crystal pattern and last year I reported on the *x*-ray examination of this material showing that it has zircon type structure (Pabst, 1951). Since then Bonatti and Gallitelli (1951) have described a non-metamict thorite differing only in having slightly smaller cell dimensions.

Discovery of the dimorphism of thorium silicate suggested that a study of the effects of heat upon thorites might be of special interest. Accordingly heating experiments have been made on seven thorites, an artificial mixture of thorianite (ThO_2) and amorphous silica in one to one proportion, and on two partly metamict zircons. The results have shown that the effects of simple heating on metamict materials may be much more complex than has been previously reported, and that the differences between various samples of the same mineral may be great.

To obtain a maximum of information from a minimum of material and observations the single crystal method was used in large part. Minute single crystals were repeatedly heated and set up for *x*-ray diffraction after each heating to allow the detection of the cumulative effects. Fragments of single crystals suitable for use on the Berman balance were employed in groups. By testing before and after heating it was, in some cases, possible to determine density, weight loss and shrinkage on the pieces used for *x*-ray study.

The only non-metamict thorium silicate available for study was the uranothorite from New Zealand reported on last year. At that time it was pointed out that heating to 850° C. has only the effect of slightly improving the *x*-ray "reflections." The same crystal heated stepwise to about 1400° C. and another crystal similarly treated showed no further changes. In each case a certain amount of disorientation remained. Attempts to use fluxes to promote a change on heating were unsuccessful due to the minuteness (less than 0.02 mg.) of the crystals.

Slightly larger single crystals of thorite from La Grange, California, described by George (1951), were found to be almost wholly metamict, yielding only a few faint powder halos with no suggestion of orientation. Heating to 760° for 15 hours entirely obliterated these traces of structure and produced no recrystallization. Further heating for similar periods at 1000° and at about 1400° C. brought first the beginning of crystallization of monoclinic $ThSiO_4$ and then the complete powder pattern of the same with no indication of other phases.

The two extreme cases just described, with no suggestion of any phases except tetragonal $ThSiO_4$ in one and only the monoclinic phase produced in the other, are the simplest found thus far. Five other metamict thorites have been examined. Essential data on the specimens are given in Table 1. Spectrographic examination showed that the minor constituents do not differ greatly and are very small in amount in most cases.

Table 2 collects some of the results obtained by heating. To the periods of heating indicated may be added the time required to heat up the furnaces, usually an hour or so, and a similar period of cooling. In all cases recorded in the table it seems certain that sufficient time was allowed to achieve essential completion of changes at a given temperature. Comparing the runs at 710, 715, 850 and 935° it is clear that shorter periods might in some cases suffice to bring about the recorded changes. It was, however, determined that changes are by no means instantaneous. Specimens *A* and *D* were heated to 850° C. for $\frac{1}{4}$ hour being placed in the fully heated furnace and air quenched at the end of the run. The results of these runs were very different from those obtained by longer heating at the same temperature. Both materials showed complete

TABLE 1. MATERIALS USED IN HEATING EXPERIMENTS

	A	*B*	*C*	*D*	*E*
Name on label	Thorite	Thorite	Orangeite	Thorite with 9% U	Thorite
Locality	"Brevig, Norway"*	Arøy, Langesundfjord, Norway*	Arö, near Langesund*	"Central Norway—Telemarken"†	Madagascar
Dealer or collector	R. M. Wilke	Minerals Unlimited	A. C. Lawson	received by A. C. Lawson from "Dahl" 1888	Minerals Unlimited
Description	loose resinous fragments	crystal embedded in nepheline syenite	amorphous fragment	waxy mass attached to orthoclase	loose crystal ca. 0.3 gram
Initial density	4.4	4.28	5.45	4.1	4.57
Color	dark brown	varying black to amber	deep orange with a few brown veinlets	uniform brown	orange, somewhat dull
Refractive index	1.705	1.664	variable 1.71–1.78	variable 1.72–1.78	1.695
Results of spectrographic examination‡					
Large amount	Th, Si	Th, Si	Th, Si	Th, Si	Th, Si
Small amount X%	—	Ca	—	Fe	—
Very small 0.X%	Ca, Fe, Mn	Na, B, Fe	Ca, Fe	Ca, Mg, Mn	Ca, Fe
Traces 0.0X–0.000X%	Li, Na, Mg, Al, B, Pb, Ti	Li, Mg, Al, Mn, Pb, Ti	Li, Na, Mg, Al, B, Mn, Pb, Ti	Li, Na, Ba, Al, B, Pb, Ti	Li, Na, Mg, Al, Pb, Ti

* Specimens *A*, *B* and *C* are all from the post-Devonian nepheline syenite pegmatite area around the Langesundfjord.

† The label of specimen *D* leaves some doubt as to its provenance. It is probably from the region of pre-Cambrian granite pegmatites near Arendal. This is within the province of Telemarken.

‡ Spectrographic examinations were made by Dr. T. G. Kennard of Kennard & Drake, Los Angeles. Uranium was tested for by fluorescence of a NaF bead. This showed specimen *B* to be lower in uranium content than the others.

but diffuse ThO_2 patterns with some lines of tetragonal $ThSiO_4$ being also present in the pattern of specimen *A*.

Only a small selection of optical observations is given in Table 2. In the main only aggregate effects are observable though inhomogeneity is easily recognizable under the microscope. In no case are crystals of sufficient size produced so that complete determination of the optical properties is possible. An increase in indices can be observed even after moderate heating. Some materials remain so fine-grained or so crowded with opaque inclusions even after indices have risen to 1.8 or more that no birefringence is discernible. Materials which are essentially tetragonal or monoclinic $ThSiO_4$ show average indices in the appropriate range. The

TABLE 2. RESULTS OF HEATING EXPERIMENTS

	A Thorite, Brevig				*B* Thorite, Arøy				*C* Orangeite, Arö				*D* Thorite, 9% U, Telemarken				*E* Orangeite, Madagascar			
	Iso. ThO_2	Tet. $ThSiO_4$	Monoc. $ThSiO_4$	Other lines	Iso. ThO_2	Tet. $ThSiO_4$	Monoc. $ThSiO_4$	Other lines	Iso. ThO_2	Tet. $ThSiO_4$	Monoc. $ThSiO_4$	Other lines	Iso. ThO_2	Tet. $ThSiO_4$	Monoc. $ThSiO_4$	Other lines	Iso. ThO_2	Tet. $ThSiO_4$	Monoc. $ThSiO_4$	Other lines
Initially	amorphous				amorphous				amorphous				amorphous				—	!d	—	—
After 40 h. at 640° C.	tr.	tr.+	—	halo 6.5 Å	tr.	—	tr.+	—	—	!	—	—	tr.	tr.	—	—	—	!d+	—	—
20 710	!	(?)	—	halo 6.1 Å					!d	!	—	—	!	tr.	—	halo 6.5 Å				
141 715	!	tr.	—	fog 6.3 Å					!d	!	tr.	—	!	15%	15%	halo 6.4 Å				
1½ 850	tr.	60%	40%	5.8 Å					!d	!	tr.+	halo 6.4	tr.	20	70	5.9 Å				
40 935	tr.	40	60	4.8 Å					—	65%	35%	—	?	20	60	5.9 Å				
14 950					tr.	—	80%	—									!	!	tr.	—
1 ca. 1200	tr.	15	85	5.8 Å					—	35	65	—	—	tr.	95	5.9 Å				
15 ca. 1400	tr.	—	95+	—	tr.+	—	95	8.6 Å	—	—	100	—	tr.+	?	75	#	!	—	!	—
Density Before After	 4.4 5.1		 (850°)*		 4.28 4.2		 (640°)		 5.45 5.58		 (850°)		 4.1				 4.57			
R. I. Before After	 1.705 1.80–1.81+		 (850°)		 1.664 1.725		 (640°)		 1.71–1.78 1.80		 (850°)		 1.72–1.78 1.77–1.82+		 (850°)		 1.695 1.739		 (640°)	
Maximum observed Density Weight loss Shrinkage	 5.26 22% 21½%		 (935°) (ca. 1400°) (635°)		 5.6 14% 33%		 (950°)† (950°) (950°)		 7.23 9.8% 32.6%		 (ca. 1400°) (ca. 1400°) (ca. 1400°)		 5.33 12.5% 26.7%		 (715°) (935°) (710°)		 6.69 16% 24%		 (ca. 140) (ca. 1400°) (640°)	

! Means that *all* lines of the indicated material were observed. d means lines diffuse.

# Black slaggy mass stuck to crucible. After cleaning with $K_2S_2O_7$ yellow residue gave nearly pure monoclinic $ThSiO_4$ pattern.

* Figures in brackets indicate temperature of heating after which measurements were made.

† No data available for higher temperatures for specimen *B*.

lower indices of many materials are probably attributable to a large proportion of amorphous substance. These materials may also contain isometric ThO_2 but this is so fine-grained that it appears merely as a dust in the amorphous groundmass.

Though behavior of the several specimens varies widely there is one common feature. All are eventually converted to the monoclinic phase. This begins to appear after prolonged heating at 715° in two cases. It is the principal phase present after heating to 935 or 950° in most cases. The tetragonal phase does not persist at the highest temperature employed. Isometric ThO_2 always appears at intermediate stages, though in one case only in small amount, and may persist to high temperature. Its presence in abundance implies the presence of free silica. In no case is there any indication of the crystalline phases of silica. Some amorphous material must persist to fairly high temperatures. No other phases were found. The halos or weak extra lines near 6 Å may be associated with the "amorphous" material.

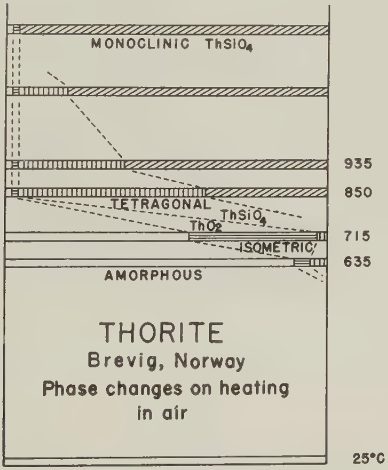

FIG. 1. Diagram of changes observed in specimen *A*.

The sequence of changes may be represented by a diagram such as Fig. 1 showing roughly the proportions of the several phases in the course of heating thorite from Brevig. A selection of the patterns indicating the phase changes for this material in four stages is shown in Fig. 2. It is apparent that the changes are very easily recognizable in this case.

These powder patterns, of course, do not allow us to see whether any single crystals or partly oriented aggregates have been present in the material. In Fig. 3 are shown two patterns of orangeites prepared from

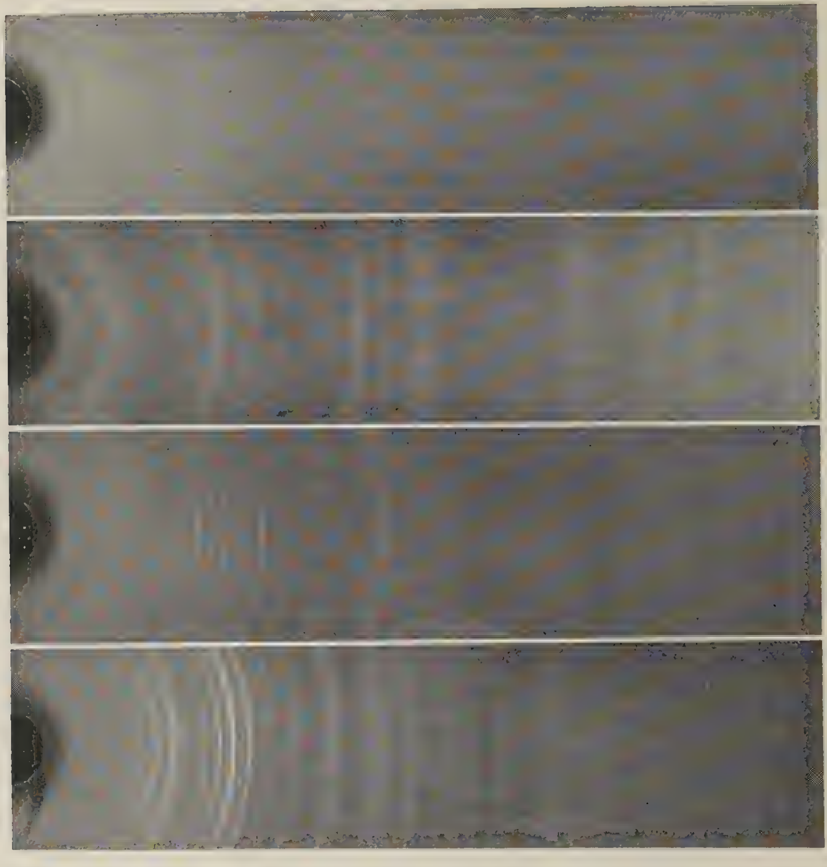

FIG. 2. Thorite, Brevig, Norway. 1. Unheated. Fully metamict. 2. Heated to 710° C. Isometric ThO_2 and amorphous. 3. Heated to 935° C. Mixture, mostly tetragonal $ThSiO_4$. 4. Heated to about 1400° C. Almost entirely monoclinic $ThSiO_4$.

single fragments. The Madagascar material yields a diffuse powder pattern even without heating. Apparently the single crystal has broken down to an aggregate or such an aggregate has formed by natural regeneration. In the Norwegian orangeite which is wholly amorphous as found there is no suggestion of orientation in the crystalline or partly crystalline material produced by heating.

Since several of the materials treated went through a stage in which they contained substantial quantities of ThO_2 with no crystalline SiO_2, but all ended up as essentially $ThSiO_4$, it seemed probable that one might synthesize $ThSiO_4$ by solid state reaction between crystalline ThO_2 and amorphous SiO_2. This was tested by making a tablet of finely ground Ceylon thorianite and amorphous silica (Baker's "Acid Silicic") intimately mixed in suitable proportion and compressed at 10,000 lbs.

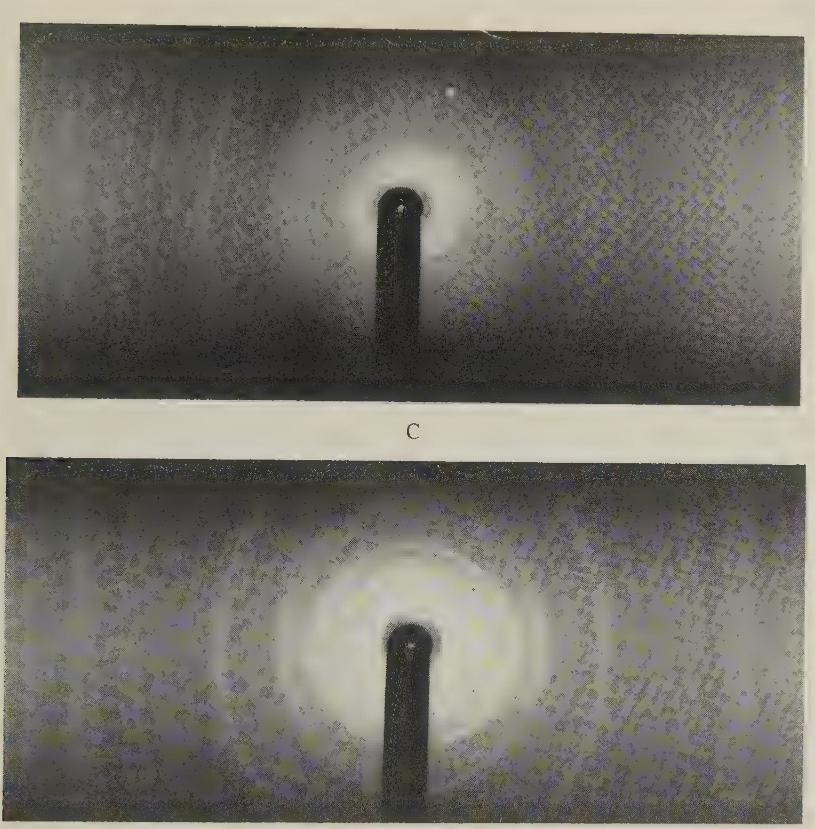

FIG. 3. *C*. Orangeite, Arö, near Langesund, Norway. Heated 40 hours at 650° C. "Single crystal" rotation pattern. Tetragonal $ThSiO_4$ only. *E*. Orangeite, Madagascar. Unheated. "Single crystal" rotation pattern. Tetragonal $ThSiO_4$ only.

per sq. in. This tablet was subjected to successive heatings. A selection of powder patterns of the products is shown in Fig. 4. The end product, as expected, is the monoclinic phase, but little of the tetragonal phase is formed at intermediate stages, possibly because reaction does not occur as readily in this artificial mixture as in metamict substances and is not extensive until the temperature range of formation of the monoclinic phase is reached.

Figure 5 shows that the monoclinic phases formed by heating thorite or by solid state reaction of thorianite and amorphous silica correspond to huttonite. The powder patterns agree in every detail.

Similar results upon heating thorites have been obtained by Mr. Kaiman (1951) of the Department of Mines and Technical Surveys, Ottawa, and by Mr. Berman (1951*a*) of the United States Geological

Survey. There remains another point to be considered. Bannister and Horne (1950) have shown that the results of heating a metamict mineral may differ with heating in air or in a neutral atmosphere. The materia they examined contained ferrous iron and its oxidation might be presumed to affect the results. Significant changes due to oxidation do not seem likely in the heating of metamict thorites. Even so it would be desirable to make tests in a neutral atmosphere. This has been done by Mr. Berman (1951). As mentioned before, he also obtained the monoclinic phase by suitable heating in air but he states "when heated in a *water free* inert atmosphere they 'revert' to the tetragonal variety at a temperature around 1200° C. Likewise, crystalline thorite will form the monoclinic $ThSiO_4$ when heated high enough in air but does not appear to change when heated in the water free helium at our maximum temperature (1300° C.)." It will be recalled that the New Zealand uranothorite in single crystals was unchanged upon prolonged heating even well above the maximum temperature employed by Mr. Berman so that it seems that other factors, for instance the perfection of the structure subjected to heating, will affect, at least, the rate of conversion.

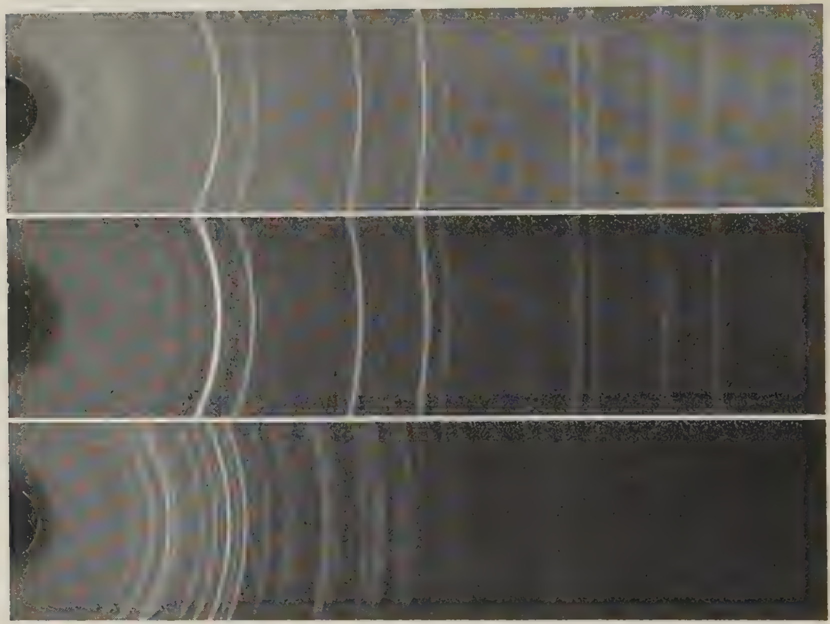

FIG. 4. Top: Thorianite, Ceylon. Middle: Thorianite and amorphous silica, after 40 hours near 1000° C. Bottom: Thorianite and amorphous silica, plus 15 hours near 1400° C.

Still another factor may influence the results of heating metamict minerals. The isotropization is in most cases attended by a certain amount of hydration. Accompanying or superimposed upon this change there may be further chemical alteration. Examination of metamict brannerite from the type locality in Idaho has shown that heating produces two kinds of cryptocrystalline material the grains of which can be distinguished by external features. Presumably one kind corresponds to the heating product of simply metamict brannerite and the other to the heating product of brannerite that has in addition suffered some chemical change while remaining in the metamict state. The point will be further investigated.

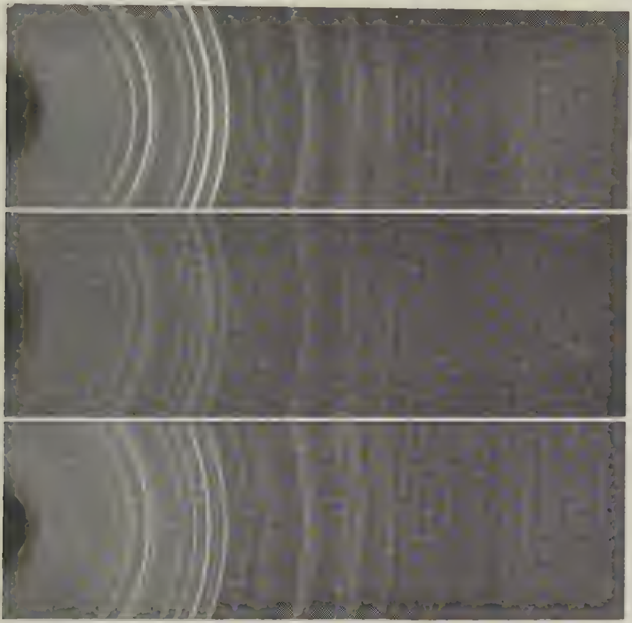

Fig. 5. Huttonite (top). Gillespie's Beach, South Westland, New Zealand. "Huttonite" (middle). Metamict thorite, Brevig, Norway, heated to about 1400° C. for 15 hours. "Huttonite" (bottom). Thorianite and amorphous silica heated to about 1400° C. for 15 hours.

Observations on Zircon

That apparent single crystals may be a complex of phases was shown by Mügge (1922) who explained the changing optical properties of gadolinites as due to a web of crystalline material within the meshes of which is enclosed an amorphous phase. The complex of phases arising upon the heat treatment of metamict zircons has been studied with care by v. Stackelberg and Chudoba and coworkers. Following the experiments on thorite I have also made a few observations on zircon using cyrtolite from Norway and zircon from Indiahoma, Oklahoma. The cyrtolite showed varying degrees of metamictness within a single crystal. At 950° C. both more and less metamict portions yielded a complete zircon pattern very diffuse at high angles with a few lines due to monoclinic ZrO_2 (baddeleyite) which persist even on heating to about 1400°, no indication of crystalline silica being found even though free silica must be presumed to be present.

The Oklahoma zircon occurs in large brown crystals that are mostly metamict. Enclosed within these are smaller crystals, some of which are fairly clear and give little suggestion of alteration. Figure 6 shows a set of rotation patterns made with such a crystal. Before heat treatment the pattern is almost that of a perfect single crystal showing little disorientation. After heating to 950° C. the same crystal yields a pattern with slightly more disorientation and showing diffraction from cubic ZrO_2 indicated by the powder arcs of this phase. After heating to about 1400° C. these powder arcs are lost but the disorientation of the parts of the crystal is even more pronounced. It is clear that here a disoriented foreign crystalline phase has arisen within the meshes of the slightly imperfect zircon host due to heating and upon further heating has disappeared while the host has become more imperfect.

ARTIFICIAL METAMICTIZATION

Since the metamict state is now generally agreed to arise from alpha particle bombardment it is surprising that so few attempts have been made to produce it artificially. Mügge (1922) was unsuccessful in attempting to alter the optical properties of orthite by exposure to radium emanation for one year. v. Stackelberg and Rottenbach (1940), on the other hand, claim to have "isotropized" a thin plate of zircon by one-sided exposure to radon for 4 months. After that period the plate cracked. This was attributed to differential expansion of the plate presumed to attend metamictization. No other indications of changes due to the irradiation were reported.

More powerful sources of radiation are now available. They might prove useful in the study of metamictization. A test case for examining the influence of structure is available in the dimorphous pair, tetragonal and monoclinic thorium silicate, which has been the principal subject of these notes. Both forms belong to well known structural types. Both have been synthesized in the laboratory of Professor Berry (1951) at Queen's University. If pure artificial preparations of these two forms were subjected to identical irradiation, using an artificial source, and differences could be established in their response, critical information would be gained on one factor in the process of metamictization.

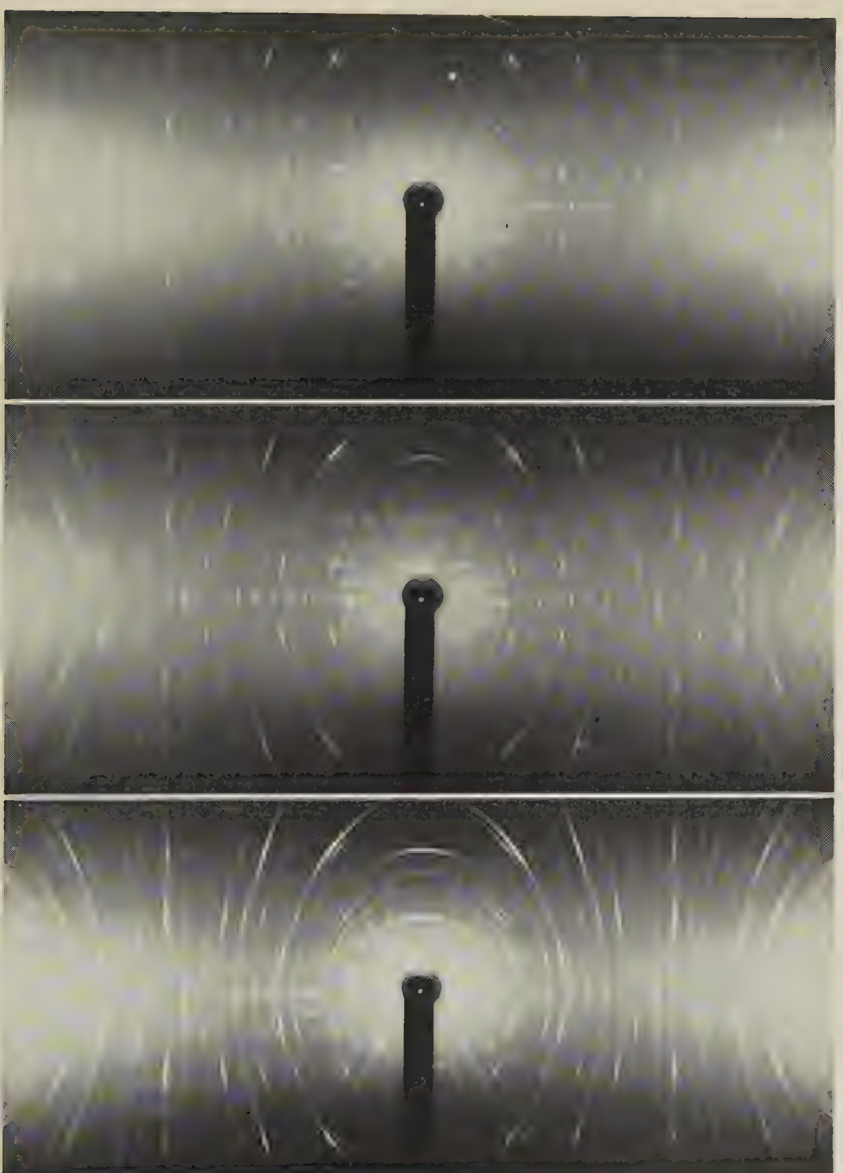

FIG. 6. Zircon, near Indiahoma, Oklahoma, *a*-axis rotation patterns. Top: Unheated. Middle: 15 hours at 950° C. Bottom: 15 hours at 950° plus 15 hours at ca. 1400° C.

ACKNOWLEDGMENTS

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MONOCHROMATOR UTILIZING THE ROTARY POWER OF QUARTZ*

C. S. Hurlbut, Jr., *Harvard University, Cambridge, Massachusetts,*

AND

John L. Rosenfeld, *Missouri School of Mines & Metallurgy, Rolla, Missouri.*

Abstract

Plane polarized light moving parallel to the *c* axis of quartz has the plane of polarization rotated various amounts depending on the wave length; the shorter the wave length, the greater the rotation. This rotary dispersion is the basis of the monochromator.

Four basal sections of quartz are used with the thicknesses in the ratios of 1:2:4:8 and so arranged that light passes successively from the thickest to the thinnest. When the polarized beam emerges from the far side of the thickest quartz, it passes through another polarizer which blocks the wave lengths vibrating at right angles to the polarization axis of this second polarizer but transmits the wave lengths vibrating parallel to that direction. In a similar way, each following stage can be thought of as eliminating half of the light entering it until finally a single band of about 150 Å width emerges. By means of mechanical gearing the turning of a single knob rotates the polarizers so that this narrow pass band can be made to move continuously from one end of the spectrum to the other.

Because of the large aperture, adequate illumination can be obtained for microscopic work by using an incandescent tungsten filament lamp.

The Emmons method of double variation is a powerful means of accurately determining the refractive index of small crystal fragments. The weakest link in the chain of apparatus that must be assembled is the monochromator. Even with using the carbon arc as a light source, the intensity of light issuing from the average monochromator is so low that prolonged work is a strain on the eyes. The development of the quartz monochromator here described was undertaken in the hope of obtaining an instrument that would give more adequate illumination for the Emmons method. The limited use to which it has already been put has given most satisfactory results and indicates that it may have a wider application than that for which it was designed originally.

Within recent years considerable attention has been given to the development of *linear birefringent filters*. Their use and construction is described by Billings (1947) and Evans (1949). Such filters are based on the interference of polarized light produced by plates of birefringent crystals. Plates are cut to give maximum birefringence, that is, parallel to the plane of the optic axes of biaxial crystals and parallel to the optic axis of uniaxial crystals. The superposition of several crystal plates of proper thickness interlaminated with parallel polarizers permits the passage of a single narrow pass band of light. Such a filter can be made to pass any predetermined wave length, or may be tuned by the insertion of quarter wave plates and rotation of the polarizers. The quartz monochromator discussed here can be described as a tunable filter utilizing the rotary power of quartz and the large rotary dispersion. Although the pass band can not be made as narrow as that of a linear birefringent filter without using a very great thickness of quartz, it has certain advantages of construction. The quarter wave plates are unnecessary and even the thinnest of the quartz plates is sufficiently thick to be easily handled by the optical worker. A quartz plate giving the same broad band by interference is exceedingly thin.

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* Contribution from the Department of Mineralogy and Petrography, Harvard University, No. 332.

TABLE 1. ROTATION OF PLANE OF POLARIZATION BY QUARTZ

λ in Å	Rotation in degrees	
	1 mm. thick	53 mm. thick
7600	12.67	671.51
6708	16.54	876.62
6563	17.32	917.96
5890	21.68	1,149.04
5461	25.70	1,362.10
5270	27.54	1,459.62
5086	29.72	1,575.16
4861	32.77	1,736.81
4340	42.60	2,257.80
4047	48.93	2,593.29
3934	52.15	2,763.95

APPROXIMATE LIMITS OF VISIBILITY

λ in Å	Rotation in degrees	
	1 mm.	53 mm.
4000	49.0	2,597
7000	15.3	811
Difference in rotation between extremes	33.7	1,786

That the plane of polarization of light moving parallel to the *c* axis of quartz is rotated is common knowledge; the magnitude of this rotation is less well known. In Table 1 the angular rotation for the plane of polarization in quartz is given for various wave lengths in the visible spectrum. Figure 2 shows graphically the relation of wave length to rotation. It will be noted that the difference in angular rotation between the extreme wave lengths (4000 Å–49°, 7000 Å–15° per millimeter of thickness) is 33.7° for a one-millimeter plate and approximately 1800° for a 53 millimeter plate. The 53-millimeter plate is considered since it was used in the construction of the monochromator.

If light is passed through a basal section of quartz, placed between polarizers, those wave lengths whose planes of polarization have been rotated so that they are parallel to the vibration direction of the upper polarizer will be transmitted. Wave lengths whose planes of vibration have been rotated so that they are at right angles to the vibration direction of the upper polarizer will be eliminated. In a spectroscope the spectrum will be found to be crossed by a number of dark bands, "channel spectra"; these bands correspond to the wave lengths rejected by the upper polarizer. In Fig. 3 is shown diagrammaticaly a band that is passed (*a*) and a band that is rejected (*b*).

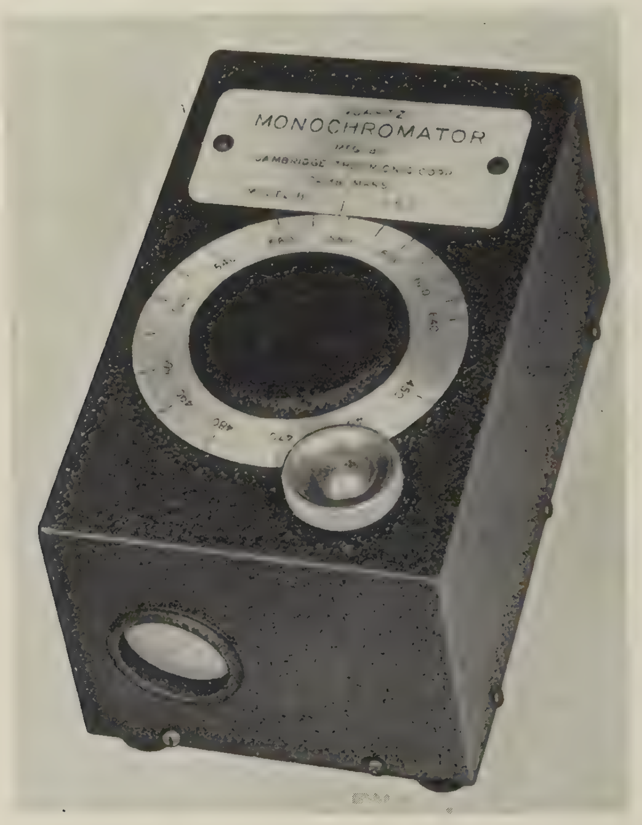

Fig. 1. Quartz monochromator.

In construction of the instrument, four basal sections of quartz are used and arranged in tandem, each section being half as thick as the one preceding. If the thickest section, #1, has unit thickness, section #2 has a thickness of $\frac{1}{2}$, #3 a thickness of $\frac{1}{4}$, and #4 a thickness of $\frac{1}{8}$. Five polarizers are used. One is to polarize the light before entering section #1 and one for each of the four quartz sections to select given wave lengths as the light emerges.

When the bands which are transmitted by the polarizer of section #1 pass through section #2, they are rotated just half as much as in the first section. Therefore, if the polarizer of section #2 is oriented properly, it will transmit alternate light bands passed by the first polarizer. For example, if two wave lengths are rotated 1200° and 1380° respectively by the first quartz section, the #1 polarizer can be set to pass both. However, on entering section #2 each is rotated half as much as previously or 600° and 690°, respectively. Consequently, when #2 polarizer is set to pass one of these, it will elminate the other. Ideally, then, the first quartz section should be just thick enough to give a difference in rotation between the extreme wave lengths of visible light of 1440°. Thus eight bands, one every 180° of rotation, would be transmitted, whereas those 90° removed would be eliminated. In this way, with correct orientation of the polarizers, four bands would pass polarizer #2, two would pass polarizer #3, and one would pass polarizer #4. Figure 4 shows transmis-

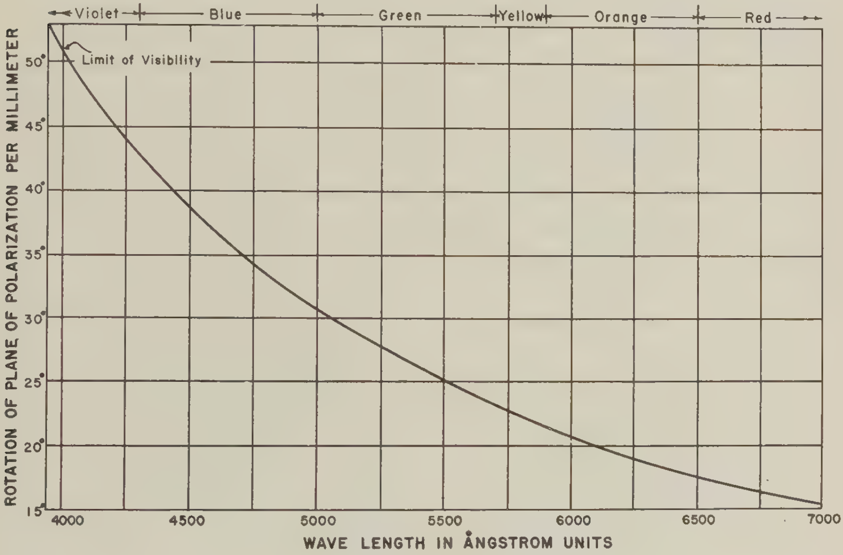

FIG. 2. Rotary dispersion in quartz.

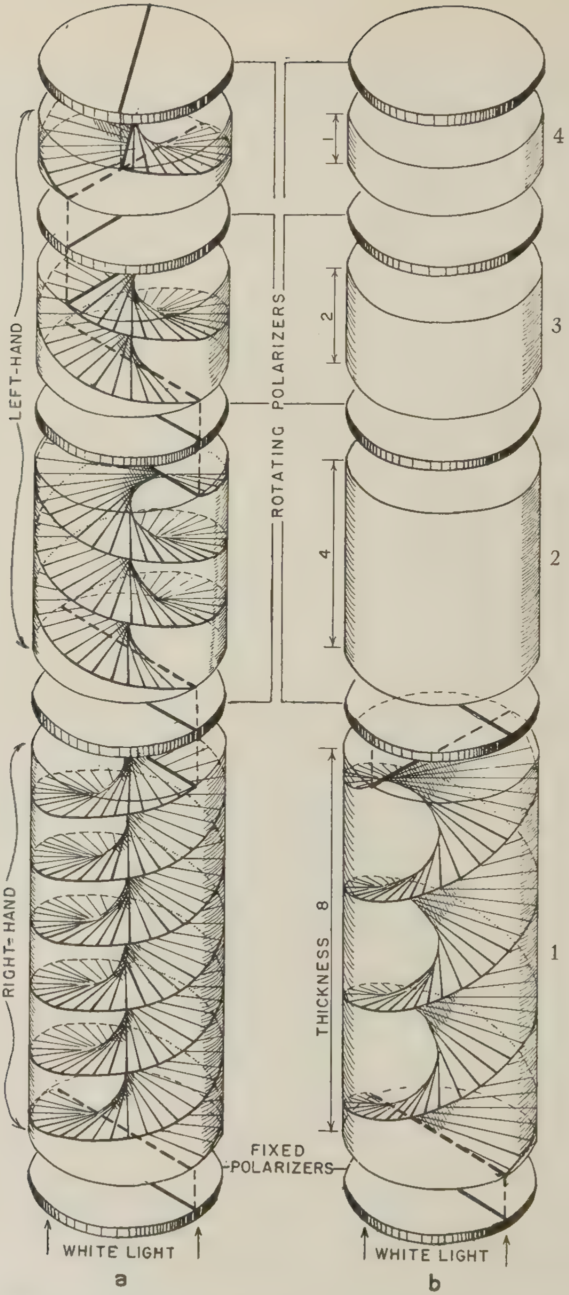

FIG. 3. Arrangement of quartz and polarizers. (a) Diagrammatic representation of the rotation of a single wave length with polarizers set to permit it to pass through the instrument. (b) Diagrammatic representation of a wave length blocked after passing through the thickest quartz section.

sion curves for four such basal sections. This arrangement is very much like a linear birefringent filter with a setting arranged to transmit a single narrow band.

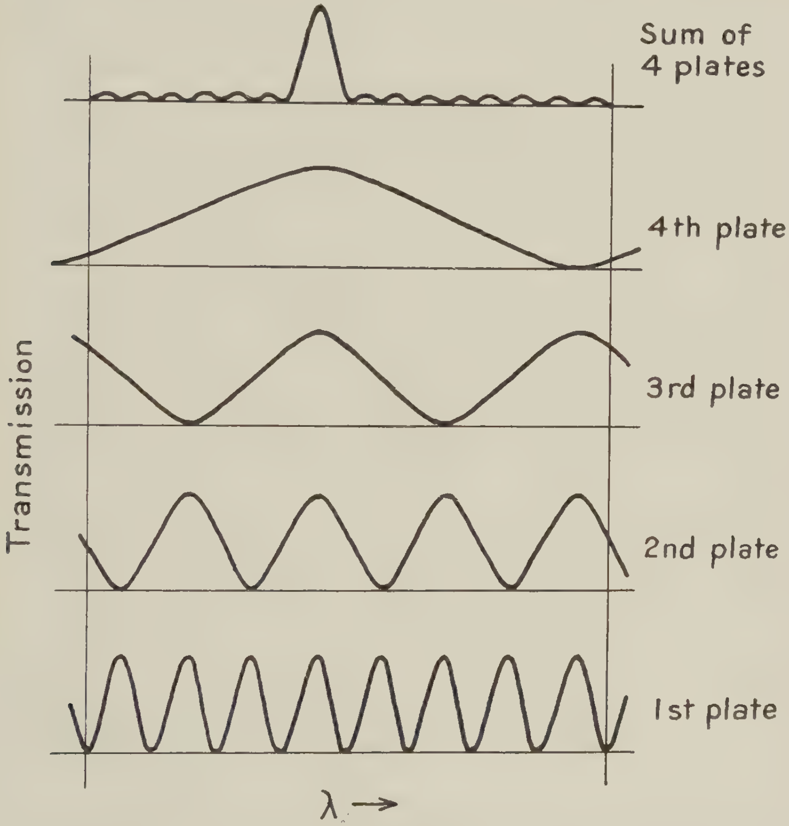

FIG. 4. Transmission curves of four quartz sections between polarizers showing the coincidence of the transmission maxima at a given wave length.

The quartz monochromator is tunable and can be set to pass a single band anywhere in the visible spectrum if the polarizers are simultaneously rotated appropriate amounts. The rotation of each polarizer is directly proportional to the thickness of the quartz plate with which it is associated. Since the thickest quartz section, #1, is twice as thick as section #2, four times as thick as section #3, and eight times as thick as section #4, the relative rotations of the polarizers are 8, 4, 2, and 1, respectively. It is possible to construct the instrument using all right-hand or all left-hand quartz by having light move from the fixed polarizer into the thinnest section.

If several instruments are to be made, it is difficult to standardize on either hand of quartz, for in nature, or in any purchased lot of crystals, about 50 per cent is right-hand and 50 per cent is left-hand. It is possible to utilize about equal amounts of both types if the thickest quartz section is of one hand and the other quartz sections of the opposite hand. This arrangement is satisfactory if light passes from the fixed polarizer into the thickest quartz section. The monochromator was constructed with the thickest section of right-hand quartz and the others of left-hand quartz (Fig. 3*a*).

When the instrument is assembled all the polarizers are set in parallel position. Mechanical gearing to a single shaft maintains the proper rotation ratios and permits the polarizers to be rotated simultaneously by turning a single knob. For example, consider a given wave length that is rotated to the right 1000° by quartz plate #1. Polarizer #1 is rotated 1000° to the right to be in a position to let it pass. On passing through quartz plate #2 this wave length would be rotated 500° to the left; polarizer #2 is rotated 500° to the right and is in a position to let it pass. Quartz section #3 will rotate the same wave length 250° to the left and polarizer #3, rotated 250° to the right, is in a position to let it pass. Finally, on passing through quartz #4, the light will be rotated 125° to the left, giving a total left-hand rotation by 2, 3, and 4 of 875°. Thus if polarizer #4 is rotated 125° to the right, it will pass the desired wave length and other wave lengths will have been effectively eliminated.

If one wishes to shift to a slightly shorter wave length, for example, one which is rotated 1020° by quartz #1, it would be accomplished by a further 20° rotation of polarizer #1 and proportionate rotations of the other polarizers. In such a way, one can move the pass band continuously from one end of the visible spectrum to the other and the wave length can be read directly on a dial.

In the preceding paragraphs the rotation of the polarizers alone has been considered. It has been found, however, that it is more practical to cement a polarizer directly to each quartz section and rotate the sections. In this way half the number of air-to-quartz and air-to-glass surfaces are eliminated and internal reflections are reduced.

Spectral distribution curves taken at seven settings show the transmission throughout the visible spectrum to be essentially uniform with between $2\frac{1}{2}$ and 3 per cent of any wave length passing the monochromator. This transmission compares favorably with the 5 per cent theoretically possible. The difference between the theoretical and measured transmission is due largely to internal reflections. Those reflections could be eliminated and the efficiency improved by immersing in oil. The average pass band width as determined by the spectral distribution curves is about 150 Å, which appears to be narrow enough for most microscopic work. With the addition of a fifth quartz section the average band width could be reduced to about 75 Å.

For the most efficient use of the monochromator the white light must be collimated. This can be effected by an external lens system, but for most purposes sufficient collimation is obtained by moving the light source several inches away from the instrument.

In using the monochromator as a light source for a polarizing microscope, it is necessary that the vibration direction of light leaving the instrument be parallel to the vibration direction of the lower nicol. For this reason the instrument is constructed so that light emerges through the fixed polarizer whose vibration direction is vertical.

The quartz monochromator has definite advantages over the conventional prism monochromator. The chief advantage is that, even with the apparent low efficiency, it can be used with an incandescent tungsten lamp as a light source and yield adequate illumination. The reason for this is that the large aperture, determined by the diameter of the quartz sections, of the quartz monochromator can be completely and effectively used. In the instruments constructed, the diameter of the quartz is one inch, but, by increasing the diameter to 1.41 inches, the amount of monochromatic light obtained from the instrument could be doubled.

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DIFFERENTIAL THERMAL ANALYSIS OF THE DOLOMITE DECOMPOSITION*

ROBERT A. W. HAUL AND HENDRIK HEYSTEK, *National Chemical Research Laboratory, South African Council for Scientific and Industrial Research, Pretoria.*

ABSTRACT

Samples of dolomite, calcite, and magnesite have been submitted to differential thermal analysis under various carbon dioxide pressures and the results obtained are discussed in relation to the reaction mechanism of the thermal decomposition of dolomite. Differential thermal peak temperatures are also compared with equilibrium dissociation temperatures of calcite and magnesite.

INTRODUCTION

In earlier communications in this series, the thermal decomposition of dolomite has been investigated by *x*-ray diffraction (Haul and Wilsdorf, 1951), and by studies of the exchange of carbon-13-dioxide between dolomite and gaseous carbon dioxide at elevated temperatures (Haul, Stein and Louw, 1951). As a further aid to the elucidation of the mechanism of this reaction, the technique of differential thermal analysis (*DTA*) has now been applied.

Differential thermal curves for dolomite have been described by various authors (Table 1) although merely as a means for its identification in minerals. Schwob (1947, 1950), however, concluded from the peaks observed at about 800° C. and 930° C. that the reaction takes place in two distinct stages; the first corresponding to the formation of magnesium oxide and calcite, and the second to the dissociation of calcite. In the presence of small amounts of sodium carbonate, the first peak was shifted to lower temperatures, approximating to the peak temperature observed for pure magnesite (700° C.). Schwob, therefore, suggested that the first phase of the decomposition of dolomite consists of a primary dissociation into magnesium and calcium carbonates. The magnesium carbonate formed in this way is unstable at that temperature and decomposes immediately, followed by the decomposition of the calcium carbonate at a higher temperature. The addition of alkali carbonate merely catalyses the separation of the constituent carbonates.

So far *DTA* work has been done by heating of samples only in air. It was felt, however, that further information on the decomposition of dolomite could be obtained only by investigating the samples under a range of constant carbon dioxide pressures. Investigations along these lines were already completed when Rowland and Lewis (1951) emphasized the importance of controlling the atmosphere in *DTA* work. Among various minerals investigated the above authors also compared mineral carbonates when decomposed in air and in an atmosphere of CO_2. The peaks obtained appeared to be more distinct in CO_2, and the peak temperatures were different. These results were in line with experience in this laboratory and the present paper records an extension of the work in which the effect of various carbon dioxide pressures on the decomposition of dolomite, calcite and magnesite, is illustrated in detail.

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* Communication No. 3 on the thermal decomposition of dolomite.

Apparatus and Experimental Technique

(*A*) *DTA apparatus*

The automatic *DTA* apparatus used in this laboratory has been described by Theron (1951) and consisted of:

(i) a horizontal kanthal A-wound Alundum tube furnace.
(ii) a differential thermocouple as well as a thermocouple controlling and measuring the furnace temperature, both made out of 0.5 mm. dia. Platinum-Platinum (13%) Rhodium wire.
(iii) a sample holder of two platinum micro crucibles (2 cm.^{3}) suspended in the reaction vessel by means of a chrome-nickel plate as illustrated in Fig. 1.
(iv) an automatic temperature controlling system which utilizes a motor driven variac operated by a proportioning electronic temperature controller.
(v) an automatic recording of the amplified differential voltage and the furnace temperature by a six point recording millivoltmeter.

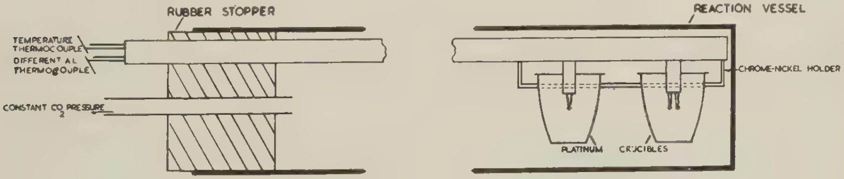

FIG. 1. Reaction vessel with *DTA* sample holder.

(*B*) *Apparatus for controlling the CO_2 pressure*

The platinum crucibles with sample and inert material (calcined Al_2O_3), together with the thermocouples, were placed in a reaction vessel of vitreous silica, 40 cm. long and 4 cm. inner dia. open at one end. This was closed by means of a rubber stopper through which were introduced an alundum tube carrying the thermocouple wires and a glass tube connected with the pressure controlling system. The thermocouple wires were sealed in at the outside end of the alundum tube with picein wax. This method, though simple, was quite satisfactory, as no leaks occurred during the experiments.

Any desired pressure up to 760 mm. (about 100 mm. above local atmospheric pressure) could be maintained constant by means of the apparatus illustrated in Fig. 2. A capillary tube (1.5 mm. inner dia.) connected with the reaction vessel was suspended inside a glass tube 1 m. long filled with mercury to the desired height and evacuated throughout the duration of an experiment. As soon as the pressure increased inside the reaction vessel due to heating or evolution of gas by decomposition of a carbonate, the gas escaped through the capillary until the original pressure determined by the mercury height was again obtained. Before starting an experiment, the reaction vessel was repeatedly evacuated and then filled with carbon dioxide dried over silica gel using the three-way stopcock *A*. During this manipulation stopcock *B* was kept open and was closed only after the last filling of the reaction vessel with carbon dioxide.

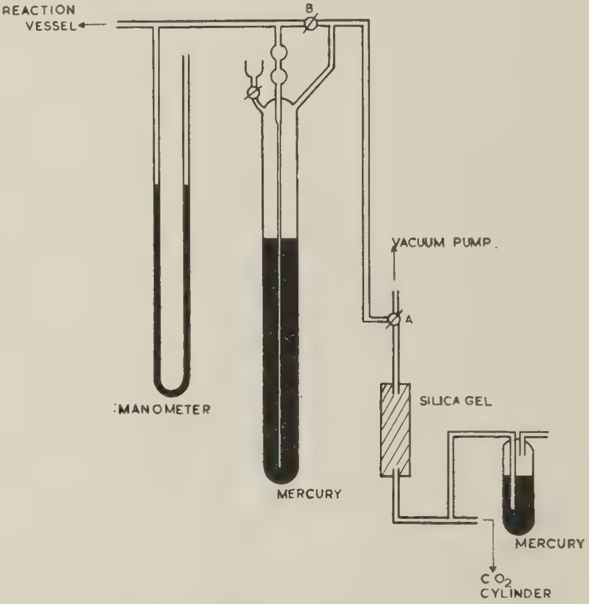

FIG. 2. Apparatus for maintaining constant CO_2 pressures.

(*C*) *Experimental technique*

Due to the vigorous gas evolution occurring when carbonates, especially magnesite, decompose at low pressures, some material was lost out of the crucible. It was, therefore, found expedient to use material of 40 to 60 mesh in preference to finely ground samples and to fasten down the crucible lid in experiments at pressures lower than 100 mm. For the same reason, in some runs magnesite was diluted using a mixture of 10% magnesite and 90% of the same magnesite previously ignited to 1000° C. In all experiments 1.5 g. of sample were used.

Normally, a *DTA* heating rate of 15° C./min. is used in this laboratory. The additional temperature lag caused by the insertion of the silica reaction vessel into the *DTA* furnace, however, made satisfactory control of the furnace temperature difficult. At a heating rate of 7° C./min. good control could be maintained and this rate was used throughout these experiments.

The inversion temperature of α, β quartz (575° C.) was used for temperature calibration. In an initial run in an air atmosphere, a peak temperature of 570° C. was obtained with a standardized thermocouple. If, however, the inversion temperature was measured under carbon dioxide pressures of 40 and 760 mm. respectively, the peak occurred at 550° C. It is thought that this discrepancy may be due to the difference in heat conductivities of air and carbon dioxide. All temperatures given in this paper were corrected accordingly.

The origins and chemical analyses of the samples used are listed in Tables 1 and 2.

RESULTS AND DISCUSSION

(1) *Comparison of differential thermal peak temperatures of various dolomites*

In Table 1 peak temperatures of dolomites recently reported in differential thermal investigations on carbonates are listed together with results obtained in the present work.

When comparing these results, and excluding runs with impure and diluted samples, it is found that the first peak maximum occurs at $810 \pm 15°$ C., and the second peak maximum at $940 \pm 25°$ C. Considering that the *DTA* apparatus used by the various authors differed to some extent, e.g. sample holder, differential thermocouple, rate of heating, and that dolomites of various purity, particle and crystal size were investigated, this agreement is surprisingly good. The somewhat bigger variation in the temperature of the second peak maximum may be explained by the dependence of this peak on the carbon dioxide pressure, as will be shown later.

In the present experiments four different dolomites were also run at a constant carbon dioxide pressure of 660 mm. The peak temperatures on comparison are close together, though the first peak of the rather impure

TABLE 1. COMPARISON OF *DTA* PEAK TEMPERATURES OF VARIOUS DOLOMITES

Samples		*DTA* technique	Atm.	First Peak temp. °C.			Second Peak temp. °C.			Remarks	Author
Source	Analysis			Beginning	Maximum	End	Beginning	Maximum	End		
Cleavage frag.	CaO :31.25% MgO:20.13% CO_2 :46.92%	Nickel block sample holder, Pt. Pt. 10% Rhod. Thermocouple 10° C./min. 60 mesh. Mixtures 75% dolom. +25% Al_2O_3	Air	600	780	(800)	(800)	830	880	peaks not completely separated	Cuthbert & Rowland (1947)
West Roxbury, Vt., U.S.A.	$\omega=1.684$ $\epsilon=1.502$	Nickel block sample holder, Chromel P. Alumel Thermocouple 12° C./min. 100/200 mesh	Air	750	815	845	855	965	985	peaks separated	Beck (1950)
Lee, Mass., U.S.A.	—	Platinum crucible, Chromel P. Alumel Thermocouple 6.7° C./min. 80 mesh	Air	760	800	830	830	950	970	peaks separated	Gruver (1950)
Rochester, N.Y., U.S.A.	with calcite			800	810	840	840	965	1000		
Pearl Spar, Joplin, Mo., U.S.A.	with magnesite			650	760	830	830	940	970		
Milville, West Va., U.S.A.	not stated	not stated	Air	—	792	—	—	926	—	peaks separated	Faust (1949)
Quillan, France	CaO :32.7% MgO:20.8% CO_2 :46.2%	Porcelain crucible divided by nickel wall	Air	780	825	(880)	(880)	940	1020	not completely separated	Schwob (1950)
Baudean, France	CaO :32.0% MgO:20.0% CO_2 :46.7%			780	800	(860)	(860)	920	1000		
Bédarieux, France, dolomite spar	CaO :30.4% MgO:21.6% CO_2 :47.8%			710	800	(860)	(860)	930	1000		

Samples		*DTA* technique	Atm.	First Peak temp. °C.			Second Peak temp. °C.			Remarks	Author
Source	Analysis			Beginning	Maximum	End	Beginning	Maximum	End		
Not stated	Not stated	Nickel block sample holder, Pt. Pt. 10% Rhod. Thermocouple 10° C./min.	Air	(750)	810	840	840	920	940	peaks nearly separated	Rowland & Lewis (1951)
			CO_2	760	790	810	910	940	960	peaks separated	
Thornwood, N.Y., U.S.A.	CaO :30.8% MgO:21.8% CO_2 :46.1%	Platinum crucible Pt. Pt. 13% Rhodium Thermocouple 7° C. /min.	Air (660 mm.)	775	825	(870)	(870)	945	995	peaks not completely separated	Haul and Heystek (1951)
			CO_2 (760 mm.)	735	795	855	890	965	990	peaks separated	
			(660 mm.)	730	790	835	885	950	975	peaks separated	
Lee, Mass., U.S.A.	CaO :30.7% MgO:21.8% CO_2 :45.8%	Platinum crucible Pt. Pt. 13% Rhodium Thermocouple 7° C. /min.	(660 mm.)	730	800	855	885	955	990	peaks separated	
Cape, South Africa	CaO :29.9% MgO:20.6% CO_2 :45.0%	Platinum crucible Pt. Pt. 13% Rhodium Thermocouple 7° C. /min.	(660 mm.)	710	770	810	890	960	995	peaks separated	
Krugersdorp, Transvaal, South Africa	CaO :28.7% MgO:19.8% CO_2 :46.0%	Platinum crucible Pt. Pt. 13% Rhodium Thermocouple 7° C. /min.	(660 mm.)	720	790	820	880	955	985	peaks separated	

Table 2. *DTA* Peak Temperatures of Dolomite, Magnesite, and Calcite Obtained at Various CO_2 Pressures

CO_2 pressure mm.	Dolomite, Thornwood, CaO:30.8%; MgO:21.8%; CO_2:46.1% peak temperatures °C.			Magnesite, S. Rhodesia, Big Dyke MgO:46.7%; CaO:0.7%; loss ign. 51.4% peak temperatures °C.			Calcite, clear cleavage frag. Cherokee County, Kansas, peak temperatures °C.		
	beginning	maximum	end	beginning	maximum	end	beginning	maximum	end
1	645	795	855	595	705	775	645	(820)	890
20	700	825	885	585	685	750	690	(860)	925
50	715	825	875	600	695	775	725	865	925
100	1. 735	790	(815)	575	725	800	755	895	940
	2. (815)	860	910						
300	1. 735	795	(835)	600	740	815	815	955	1025
	2. (835)	895	935						
660	1. 730	790	835	600	745	815	870	995	
	2. 885	950	975						
760	1. 755	810	950	595	765	825	895	1015	1055
	2. 900	975	1000						
Air	1. 775	825	(870)	(545)	765	(835)	(725)	985	1045
	2. (870)	945	995						

South African Cape dolomite is somewhat lower. The same specimens were also investigated by determining the loss of weight when heated at 100 mm. CO_2 for 150 minutes at various temperatures in the range of 550° to 850° C. The pure Thornwood, New York, and Lee, Massachusetts, dolomites gave duplicate curves, while the curves of the Cape and especially the Krugersdorp, Transvaal, dolomites differed considerably in that a gradual decomposition starting at lower temperatures was indicated. In this respect, the thermo-gravimetric method provides more information than the *DTA* method.

(2) *Influence of carbon dioxide pressure on differential thermal curves of dolomite.*

When dolomite is decomposed in air, the two peaks appearing in the *DT* curves are well separated in some experiments, while in others the second peak starts before the first is completed, as already mentioned by Rowland and Lewis (1951). These variations are due to the fact that the partial pressure of carbon dioxide increases within the sample holder when CO_2 is released during decomposition. When the above mentioned authors, in their experiments, used an atmosphere of CO_2, they found that the peaks were then well separated.

In the present investigations various, but constant, carbon dioxide pressures were maintained during decomposition, as already described. The *DT* curves obtained are reproduced in Fig. 3. The peaks, which were clearly separated at 760 mm. CO_2 pressure, gradually approach one another and finally merge as the pressure is decreased below about 100 mm. The peak temperatures are listed in Table 2, and the maximum peak temperatures are plotted in Fig. 4. From this it can be seen that the first peak due to formation of MgO and $CaCO_3$ is not at all affected by the CO_2 pressure; the peak maximum remains at about 795° C. However, the second peak maximum corresponding to the decomposition of the primarily formed calcite is shifted to higher temperatures with increasing pressure, from about 800° C. to 970° C. The curve (Fig. 4) is very similar to that obtained with pure calcite, but shows that the second peak maximum of dolomite occurs about 50° lower than the peak of mineral calcite at different CO_2 pressures. This is obviously due to smaller crystal size as well as to lattice distortion present to an appreciable degree in calcite formed from dolomite, as was found by *x*-ray study. (Haul and Wilsdorf, *loc. cit.*)

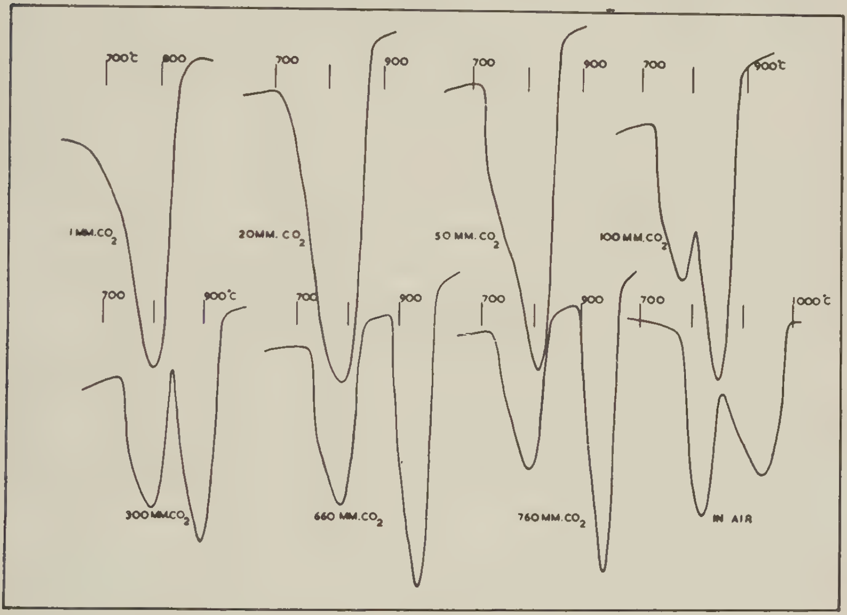

FIG. 3. Differential thermal curves of dolomite at different CO_2 pressures.

Unfortunately, with the method used, it was difficult to investigate the decomposition of dolomite at CO_2 pressures lower than about 40 mm., as some substance was always lost out of the crucible. Thus the peak temperatures at pressures in this low range may be somewhat in error. They clearly correspond, however, to the calcite decomposition.

Although it was found that the first dolomite peak is not affected by varying the CO_2 pressure, this fact is only applicable in the pressure range where dolomite is decomposed in two stages according to:

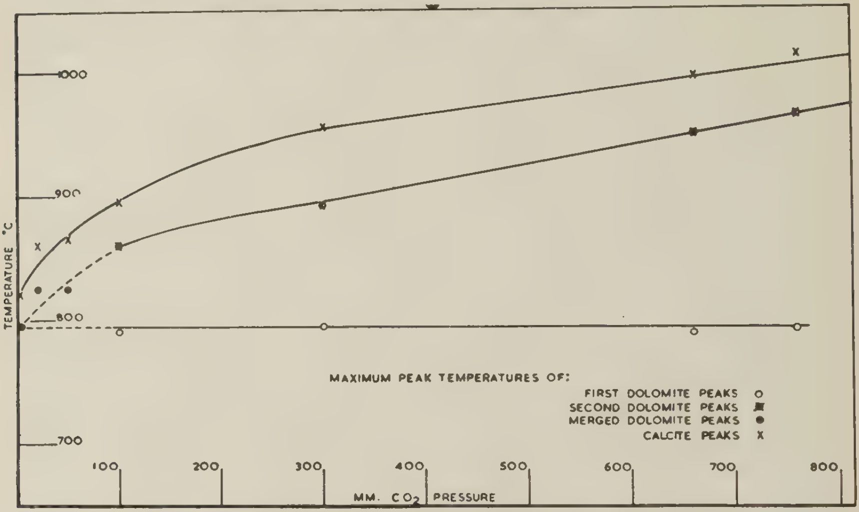

FIG. 4. Maximum differential thermal peak temperatures of dolomite and calcite at various CO_2 pressures.

$$\text{first peak:} \quad CaMg(CO_3)_2 \rightarrow CaCO_3 + MgO + CO_2 \tag{1a}$$

$$\text{second peak:} \quad CaCO_3 \rightleftarrows CaO + CO_2. \tag{1b}$$

The reaction mechanism is different when the maintained CO_2 pressure is decreased sufficiently below the dissociation pressure of calcite. This is evident from a kinetic investigation of the decomposition of dolomite by Bischoff (1950), as well as from thermo-gravimetric experiments carried out by Noll (1951) and in this laboratory.* Under these conditions, the decomposition occurs instantaneously in one stage.

$$MgCa(CO_3)_2 \rightarrow CaO + MgO + 2CO_2. \tag{2}$$

For this reason the *DTA* results are of significance only in connection with the reaction mechanism involved in the partial decomposition to MgO and $CaCO_3$. Haul and Wilsdorf (1951) have discussed various mechanisms suggested for this decomposition in the light of their *x*-ray study of thermally decomposed single crystals of dolomite and have shown that the following mechanisms do not apply:

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* To be published elsewhere.

(i) Formation of solid solutions of carbonates or oxides during decomposition (Baikov 1913; Mitchell 1923; Gel'd and Esin 1949.)
(ii) Primary dissociation into separate carbonates followed by their independent decomposition (Potapenko 1932; Conley 1939; Fagueret 1940; Schwob 1947, 1950.)
(iii) Primary dissociation into oxides and recombination of CaO with CO_2 from the gas phase to calcite (discussed by Flood 1950).

An interpretation of the first differential thermal peak as due to the "decomposition of the $MgCO_3$ part of the dolomite structure" (Beck 1950) is irrelevant. In the crystal structure of dolomite it is implicit that it must be treated as a definite compound when considering the thermal decomposition.

From the *x*-ray results, it was concluded that calcite is directly formed from the original dolomite lattice. This was indicated by the fact that calcite was present as crystallites oriented in exactly the same way as the original dolomite, thus resulting in an *x*-ray diagram equivalent to that of a calcite single crystal. The transition of dolomite into the isotype calcite lattice can easily occur, as only interchange of magnesium and calcium ions and expansion of the lattice to less than 4% are necessary. Due to the difference in crystal structure and lack of suitable corresponding atomic arrangements, the magnesium oxide crystals are not oriented to the original dolomite crystal, but grow from nuclei formed at random when a sufficient excess of magnesium ions has accumulated.

While the dependence of the second peak on the CO_2 pressure can easily be understood from basic thermodynamics, the fact that the first peak is not affected requires some explanation. As the change in free energy of the formation of dolomite from magnesite and calcite is rather small (1.6 kcal. Roth, 1936) the dissociation pressure thermo-dynamically calculated for a reaction according to equation (1*a*) will be slightly less than that for magnesite. This pressure at e.g. 800° C. is 7500 atm. Therefore in the temperature range of the first dolomite peak, the theoretical dissociation pressure exceeds the carbon-dioxide pressure maintained in the experiments by far. Consequently, the first stage of dolomite decomposition should occur at considerably lower temperatures than experimentally observed. However, the rate of this solid state reaction is determined by diffusion of the components within the lattice. It is well known that the rate of these activated processes is exponentially dependent on the temperature, i.e.

$$D = D_0 C^{-\lambda/RT}$$

where D is the average diffusion coefficient; D_0 a constant and λ the activation energy. Therefore, the lattice constituents can be expected to reach sufficient mobility for the reaction (1*a*) to proceed in a rather narrow temperature range.

These considerations explain the experimental finding that the first stage of the dolomite decomposition is independent of the CO_2 pressure and always occurs within the same narrow temperature range.

(3) *Comparison between differential thermal peak temperatures and equilibrium dissociation temperatures of calcite and magnesite.*

The equilibrium constant of the thermal decomposition of carbonates

$$MeCO_3 \rightleftarrows MeO + CO_2$$

as is well known, may be written

$$K_p = \frac{p_{CO_2} \times p_{MeO}}{p_{MeCO_3}} \equiv p_{CO_2}$$

and furthermore the relation between partial pressure of carbon dioxide and dissociation temperature is given by

$$\log p_{CO_2} = -\frac{H_T}{4.57 \cdot T} + \frac{S_T}{4.57}$$

where H_T and S_T represent the differences in heat content and entropy for the initial and final products at the temperature T for 1 Mol. By means of this equation, the thermodynamic equilibrium pressures can be calculated for various temperatures and these values are plotted in Fig. 5. While for calcite there is quite good agreement between the experimental determined (Johnston, 1910) and calculated values (Margulesco, 1946), for magnesite most investigators have recorded pressures far less than those corresponding to equilibrium. Only recently has Cremer (1949) succeeded, by the use of special experimental conditions, in getting data near the thermodynamically calculated values.

When comparing the differential thermal peak maximum temperatures for calcite with the equilibrium values (Fig. 5.) at different CO_2 pressures, the former temperatures are about 120° higher. However, if the temperatures at which the peaks start are compared, a fairly good agreement is obtained, though the measurement of this point on the *DT* curve is sometimes difficult. Therefore, the temperatures of the beginning of the peaks are more significant as far as the equilibrium temperature is concerned. The peak maximum temperatures are more affected by the conditions of heat conductivity, particle size, etc. For mineralogical analysis, of course, the commonly used peak maximum is sufficient and also more convenient to measure.

When considering magnesite (Fig. 5), it is found that the peak maximum temperatures exceed the corresponding equilibrium temperatures by about 400° C. Even when magnesite is diluted with magnesium oxide (1:9) the peak maximum is still about 300° C. too high. This discrepancy is due to the fact that the rate of diffusion processes within the solids determines the temperature range where the reaction occurs, as already discussed in connection with the mechanism of the first stage of dolomite decomposition. According to this, there should also be no dependence of the peak temperature on the CO_2 pressure. The experimental values, however, indicated a small difference (35 degrees) between the maximum differential thermal peak temperatures at 40 and 760 mm. CO_2 respectively. When the temperatures of the beginning of the peaks are considered, no dependence on CO_2 pressure is observed (Fig. 5). The slight influence of different CO_2 pressures on the maximum temperature of the

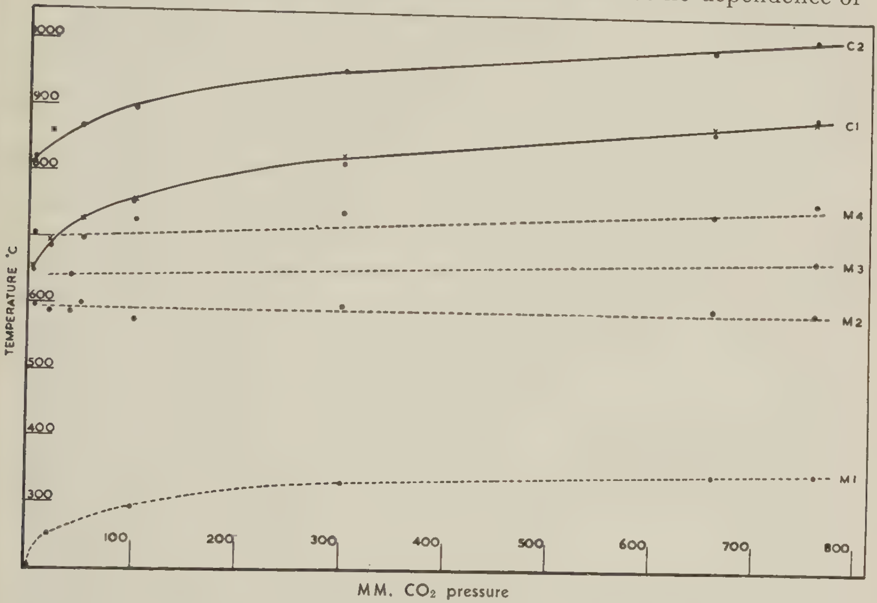

FIG. 5. Comparison of *DT* peak temperatures of calcite and magnesite with equilibrium dissociation temperatures:

- C_1—equilibrium dissociation temperatures for calcite,
- ·—temperatures of the beginning of calcite peaks,
- C_2—maximum temperatures of calcite peaks,
- M_1—equilibrium dissociation temperatures for magnesite,
- M_2—temperatures of the beginning of magnesite peaks,
- M_3—maximum peak temperatures for magnesite diluted with magnesium oxide (1:9),
- M_4—maximum peak temperatures for magnesite.

differential thermal peaks may be due to variations in heat conductivity and diffusion of carbon dioxide through the sample.

CONCLUSIONS

(1) Comparison of various dolomites has shown that although different apparatus and techniques have been used by different authors, the *DTA* peak temperatures obtained are in reasonable agreement. The first peak occurs at 810±15° C. and the second at 940±25° C.

(2) A variation in carbon dioxide pressure in the range 760 to 0.5 mm. Hg resulted in a lowering of the second peak temperature of the dolomite decomposition with decreasing pressure while the first peak was not influenced. Below about 100 mm. CO_2 the two peaks merged.

(3) These results are in agreement with a reaction mechanism of the thermal decomposition of dolomite derived from a previous *x*-ray study. As far as decomposition in two stages is concerned, the calcite in the first stage is directly formed from the isotype dolomite structure by the diffusion of its constituents within the lattice. The rate of these activated diffusion processes and not the thermodynamic given dissociation pressure determine the temperature range when the first stage of the decomposition occurs. Therefore, the first differential thermal peak is not influenced by a variation of the carbon dioxide pressure.

(4) While the maximum peak temperatures of calcite are about 120° C. higher than the equilibrium dissociation temperatures in the above pressure range, the temperatures of the beginnings of the peaks are in fair agreement with the equilibrium temperatures. The peak temperatures of calcite decomposition increase with an increase of CO_2 pressure, as can be expected thermodynamically.

(5) The differential thermal peak temperatures of magnesite are much higher than the calculated dissociation temperatures and the beginnings of the peaks show no dependence on the CO_2 pressure. This is due to the fact that the rate of decomposition is determined by diffusion processes within the solid state.

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OCCURRENCE OF BETA TRICALCIUM PHOSPHATE IN NORTHERN MEXICO

J. G. Cady, W. L. Hill, E. V. Miller,* and R. M. Magness
U. S. Department of Agriculture, Bureau of Plant Industry,
Beltsville, Maryland.

Abstract

Beta tricalcium phosphate occurs as white to slightly greenish, hard, brittle masses with occasional chalkiness in veins up to one yard in thickness running through an earthy material, consisting of fine-grained beta tricalcium phosphate and quartz, lying in cavities in calcareous sedimentary rocks (chiefly limestone), and as pale yellow crystals, 1–2 mm. in length lining cavities in a white to yellowish brittle rock. The rhombohedra from both sources show the same indices of refraction; $\omega=1.623$ and $\epsilon=1.620$ with Na light.

A number of samples of phosphate from the Mercedes Mine,† Sabinas Hidalgo, Nuevo Leon, Mexico, were brought to the Bureau of Plant Industry, Soils and Agricultural Engineering laboratory in May 1950 for identification and analysis. The beta form of tricalcium phosphate was found to be the principal phosphate mineral in the specimens.

The natural mineral forms of tricalcium phosphate have been named martinite (3, 4) and whitlockite (2). Both have the x-ray diffraction patterns of $\beta\text{-}Ca_3(PO_4)_2$. Martinite, found in island phosphates has indices of refraction $\omega=1.607$, $\epsilon=1.604$, and contains water and CO_2. Whitlockite, a hydrothermal product associated with granite pegmatites, has indices of refraction $\omega=1.629$, $\epsilon=1.626$ and contains little water and no CO_2.

The Mexican $\beta\text{-}Ca_3(PO_4)$ occurs in cavities in calcareous sedimentary rocks, chiefly limestone; some of the limestone is thought to be phosphatic. The major part of the deposit now being mined is a brownish-red earthy material. White veins of $\beta\text{-}Ca_3(PO_4)_2$, some of large size, occur in, and at the edges of the soft earthy phosphate. The deposit has the appearance of a filled cave or solution cavity. Just above the deposit is a miniature syncline, which could have resulted from slumping after solution of the limestone, or could have antedated the phosphate and been a potential collector of solute-carrying ground water. No evidence of any high-temperature activity in the vicinity has been reported.

Description of the Specimens

1. White to yellowish, hard brittle rock, flecked with black; has cavities lined with pale-yellow crystals. Powder x-ray diffraction pat-

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* Present address: Department of Agronomy, N. C. State College of Agriculture and Engineering, Raleigh, N. C.

† The samples were collected with the cooperation of Sr. Ignacio Pesqueira, President, and the assistance of Sr. Miguel de los Santos, Mine Manager, Fertilizantes de Mexico.

terns of bulk-ground samples contain interference maxima of β-$Ca_3(PO_4)_2$, quartz and a trace of apatite. Microscopically the $Ca_3(PO_4)_2$ is in the form of rhombohedra, the apatite in banded, oriented, radial aggregates, and quartz is in both prismatic crystals and chalcedony. The $Ca_3(PO_4)_2$ crystals lining cavities are 1–2 mm. long but those in the mass of the rock are 0.01–0.05 mm. long. Indices of refraction of the $Ca_3(PO_4)_2$ for Na light are $\omega=1.623$, $\epsilon=1.620$.

2. White to slightly greenish, hard, brittle masses that are chalky in spots. The veins of this rock are said to range up to one yard in thickness. Powder x-ray diffraction patterns are almost identical with those made on a specimen of whitlockite from the type locality and with synthetic β-$Ca_3(PO_4)_2$. The mineral occurs as rhombohedra, which appear to be cemented together with an unidentifiable amorphous substance. Traces of quartz, apatite and a carbonate are present. The indices of refraction are as described above. Thin sections showed great uniformity in the rock, the only structure being a weak rhythmic banding due to slight differences in crystal size, orientation and proportion of the cementing substance. The crystals are uniform in size, ranging mainly between 0.01 and 0.04 mm. The chemical analysis is given in Table 1.

3. Red earthy material, resembling a red clay soil, is the chief mine product. It has a weak β-$Ca_3(PO_4)_2$ powder x-ray pattern, plus 15–20 per cent of quartz. It is too fine-grained and stained to be studied microscopically.

4. Hydroxyl-apatite, as white to yellow crusts over essentially amorphous gray black and brown material, is present near the $Ca_3(PO_4)_2$. The crusts consist of oriented aggregates of fibrous crystals. Its chemical analysis is given in Table 1.

The chemical composition of the tricalcium phosphate in its major constituents is close to that of both the New Hampshire whitlockite and the Curacao martinite. The high zinc content is noteworthy; zinc and magnesium substituting for calcium would bring the P_2O_5:base ratio fairly close to that of synthetic $Ca_3(PO_4)_2$. The water and CO_2 contents, particularly the latter, are low in comparison with martinite and most other $Ca_3(PO_4)_2$ reported in island phosphates. Geiger-counter x-ray spectrometer patterns of the Mexican phosphate mineral, New Hampshire whitlockite and synthetic beta $Ca_3(PO_4)_2$ were nearly identical. The indices of refraction are very close to the indices of the synthetic material, thus slightly lower than the New Hampshire specimen and considerably higher than martinite. The rhombohedral habit described (2, 3) is common to the natural materials but seldom seen in the synthetic preparations. It has been suggested (1) that the name whit-

TABLE 1. COMPOSITION OF BETA TRICALCIUM PHOSPHATE AND HYDROXYL-APATITE FROM MEXICO

Constituent	Beta $Ca_3(PO_4)_2$[a]	Hydroxyl-apatite
P_2O_5	44.28	38.61
CaO	46.35	53.36
MgO	2.17	<0.05
ZnO[b]	1.99	—
Al_2O_3	0.11	0.09
Fe_2O_3		
Na_2O (K_2O)	0.05	—
MnO	<0.01	<0.01
SiO_2[c]	2.80	—
F	0.02	0.35
CO_2	0.02	—
SO_3	0.73	0.00
$H_2O+105°$[d]	1.61[e]	2.51[f]
Apatite H_2O	—	1.63
$H_2O-105°$	0.10	3.26
Qualitative Spectrographic analysis[g]		
5%	Ca, P	Ca, P
1–5%	Mg, Zn	—
0–X%	Mn	Si, F, Zn
0–0X%	Al, Si, Fe	Mg
0.00X%	Cu, V, Sr	Mn, Cu, Sr, Ba, Fe
0.000X%	Ni, Ba	Al

[a] Composition required by formula; 45.78% P_2O_5, 54.21% CaO.

[b] Glenn Edington, *analyst*.

[c] Material insoluble in 1:1 $HCl+SiO_2$ thrown out by dehydration.

[d] Exclusive of water lost at 105° C.

[e] Ignited at 1200° C.

[f] Ignited at 1100° C.

[g] Spectrographic analysis by courtesy of Geochemistry and Petrology Branch, Geological Survey, U.S.D.I., Mrs. E. L. Hufschmidt, *Analyst.*

lockite be used for β-$Ca_3(PO_4)_2$ and martinite for the corresponding mineral containing CO_2 and H_2O. The Mexican specimen is nearly CO_2 free and contains much less H_2O than martinite and has high refractive indices but in its low-temperature origin it resembles martinite. The zinc content perhaps also may be considered in deciding upon its name. If a series of minerals exists containing assorted substituted ions in various proportions and having gradations in properties it may become difficult to decide upon names for intermediates. In strictest interpretation based on composition and origin the Mexican mineral probably should be called martinite.

The large size of this deposit, with its relatively high purity, in a locale of sedimentary rocks is of considerable mineralogical interest. The Grafton County, New Hampshire, whitlockite is a high-temperature product and all synthetic $Ca_3(PO_4)_2$ thus far prepared has been from high-temperature systems such as processes for thermal defluorination of phosphate rock. Martinite and related minerals are generally minor constituents in apatite or phosphorite rocks that contain rather large amounts of impurities. Beta tricalcium phosphate has been found as a constituent of urinary calculi (5)—a further indication that under the proper conditions it is a stable compound in solutions and probably can precipitate quite rapidly.

For $Ca_3(PO_4)_2$ to form from solution the required conditions include a solution having a high PO_4:Ca ratio plus some mechanism for preventing the formation of apatite. Slow precipitation in a slightly acid medium might provide the right environment. Organic or mineral colloids or nitrogen compounds may play a role in many cases such as in the island guano occurrences and the urinary calculi. The amorphous interstitial substance among the crystals in the Mexican specimen suggests an association of a colloidal system with the deposit. The presence of apatite mixed with the β-$Ca_3(PO_4)_2$ and the pure minerals in nearby parts of the same deposit indicates that precipitation conditions were delicately balanced and shifted from one mineral to the other at different times and places. The Mexican deposit appears to have accumulated as a product of the leaching action of ground water upon phosphatic limestone. Weathering of bat guano may have contributed, but the shape and nature of the deposit does not indicate this very strongly. Further field work and petrologic study are needed to establish definitely the history of the deposit.

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PREPARATION OF NUCLEAR-TRACK PLATES AND STRIPPING FILMS FOR THE STUDY OF RADIOACTIVE MINERALS*

L. R. Stieff and T. W. Stern,
U. S. Geological Survey, Washington, D. C.

Abstract

Some modifications of current techniques for preparing nuclear plates for mineralogical studies are described. A method of mounting nuclear-track stripping film on thin sections is given. Illustrations are presented of the application of these techniques to the study of the radioactivity of powdered rocks, minerals, and thin sections.

Introduction

Since Becquerel's discovery of radioactivity in 1896 by the exposure of an unactivated uranyl sulfate phosphor to a photographic emulsion, the photographic method has become a valuable tool in the study of radioactive minerals. Autoradiographic plates, nuclear-track plates, and autoradiographic stripping film may be used to recognize radioactive minerals and to study the distribution of radioactivity in rocks. In addition photographic emulsions have been used in many specialized mineralogic studies, not discussed in this paper, such as the semiquantitative determination of both uranium and thorium in the same rocks or minerals (Coppens, 1950, p. 21; Picciotto, 1949, p. 80; Yagoda, 1949, p. 160), the departure of uranium minerals from radioactive equilibrium (Yagoda, 1949, pp. 101, 182), the artificial autoradiography of minerals (Goodman and Thompson, 1943, p. 456), and the natural fission rates and neutron fluxes in uranium ores (Yagoda and Kaplan, 1949, p. 702).

The autoradiographic method of studying the distribution of radioactivity in rocks is the oldest and simplest. A polished rock or mineral section is placed in contact with the emulsion of a photographic plate sensitive to either light, to *x*-rays, or to alpha particles. After the plate has been properly exposed and developed, the radioactive minerals are recognized by comparing the pattern on the photographic plate with the polished specimen. Bowie (1951, p. 65) and Poole and Bremner (1949, p. 130) using mechanical means of registering the autoradiographic plate and specimen have been able to locate radioactive grains 20 to 25 microns in diameter. If more precise registration is required, however, mechanical methods of alignment are not entirely satisfactory. The microscopic study of the superimposed alpha plate is also limited to microscope objectives which have greater working distances than the

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* Publication authorized by the Director, U. S. Geological Survey.

thickness of the glass autoradiographic plate. Because of these shortcomings and because this simple method has been fully described by Yagoda (1946*a*, p. 87) and others, no further elaboration of this technique is given herein.

The dispersed-grain method of Mügge (1909, p. 114), of Tyler and Marais (1941, p. 146), and of Yagoda (1949, p. 176) has solved the problem of registering the developed alpha or beta tracks with the radioactive grain that emitted the energized particle, by entrapping powdered rock or mineral fragments in a water-softened emulsion. This method uses special photographic plates covered with thick, fine-grained emulsions (nuclear emulsions) capable of recording the trajectories of alpha and beta particles emitted by both natural and artificial radioactive elements. Although this technique provides a means of recognizing radioactive grains, obviously it can give little information on the spatial distribution of the radioactive minerals in the rock. In spite of this drawback, this technique has many important uses, and the modifications of it developed in the Trace Elements Section, Washington Laboratory of the Geological Survey are described below.

The stripping-film method has been used in tracer studies of histological thin sections by Pelc (1947, p. 749), Boyd and Williams (1948, p. 225), MacDonald, Cobb, Solomon, and Steinberg (1949, p. 117), Berriman, Herz, and Stevens (1950, p. 472), Doniach and Pelc (1950, p. 184), and Heller (1950, p. 8), but, to our knowledge only Bowie (Personal communication, 1951) has used stripping film for geologic studies. The use of stripping film permits positive recognition of radioactive grains, and the permanent registration of the emulsion and the thin section also provides a means of studying the spatial distribution of the radioactivity in the rock. We have successfully used the method described below in mounting stripping films on more than 100 thin sections of rock.

Dispersed-Grain Method

Type of plate.—The alpha-sensitive nuclear-track plates used in the dispersed-grain method may be obtained from the Eastman Kodak Co., of Rochester, N. Y., and from Ilford Ltd., and Kodak Ltd., in England. Eastman and Ilford emulsions used for this work are designated as *NTA* and *C-2*, respectively. These plates are available in various sizes from 1 by 3 inches to 8 by 10 inches. Emulsions are available in thicknesses of 25, 50, 100, and 200 microns. An emulsion thickness of 50 microns is recommended for mineralogical work because the maximum range of the most energetic alpha particles in the emulsion does not exceed 50 microns. Sixty days should be allowed for the delivery of these plates.

For this method Kodak *NTA* plates $1\frac{1}{4}$ by 10 inches with an emulsion thickness of 50 microns were used. These plates are sensitive to light and should be handled under a $7\frac{1}{2}$-watt safelight with a Wratten series *OA* filter. The plates, emulsion side down, are cut into $1\frac{3}{4}$-inch lengths by means of a small plate-cutting jig shown in Fig. 1. A Goodell-Pratt wheel-type glass cutter is used to score the glass side of the plate. After the plate is scored, it is advanced and broken over the end of the jig.

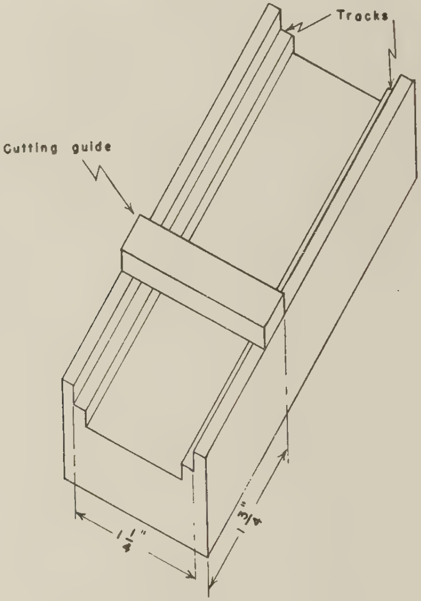

FIG. 1. Alpha-plate cutting jig.

An identifying number is then inscribed on the glass side of the plate with a diamond-pointed pencil.

Before studying rocks containing trace amounts of uranium and thorium, it may be desirable first to remove from the plate the tracks that have accumulated since the plate was manufactured. These tracks are due to traces of uranium and thorium in the emulsion. Yagoda(1946*a*, p. 94; Wiener and Yagoda, 1950, p. 39) has found that the background tracks may be eradicated by placing the plates in a light-tight container over a 3-per cent solution of hydrogen peroxide for 4 hours.

Sample preparation.—Usually specimens to be studied by this method should be ground and sized. Material of sieve sizes from minus 80 to minus 200 mesh has been used with success. If necessary, the sample may be further separated by magnetic or heavy-liquid methods and the resulting separates placed on a nuclear-track plate (Fig. 2). Also chemical separation of the sized material before dispersal is possible. After the sample is prepared, the sized fractions may be stored in a closed container for approximately a month to allow radon and its daughter products in the sample to return to radioactive equilibrium. Prior to preparing the plates, approximately 20 mg. of this sized material is dispersed in 10 ml. of distilled water in a capped 50-ml. bottle.

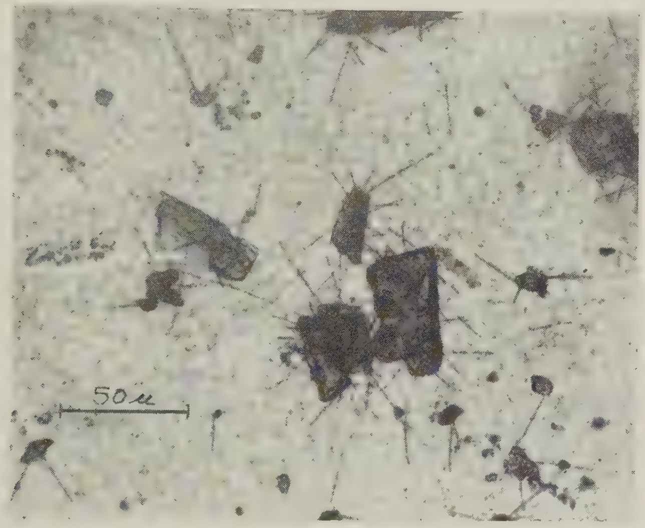

FIG. 2. Photomicrograph of crystalline carnotite from the May Day mine, Mesa County, Colo., dispersed on a nuclear-track plate. The tracks in the emulsion diverging from the carnotite grains are the paths taken by the alpha particles emitted by uranium or its radioactive daughter products. The time of alpha-plate exposure was 5 days.

Plate preparation.—A 6-cm. petri dish is filled with 10 ml. of distilled water at room temperature. The $1\frac{3}{4}$-inch alpha plate is placed, emulsion side up, in the petri dish and allowed to soak for 5 minutes. The emulsion has then softened sufficiently to trap the dispersed material which now is added near the edge of the petri dish. Care must be taken not to pour the dispersed material directly over the plate, as this loads it unevenly. The plate will now be covered with approximately 8 mm. of water. The time required for the sample to settle out depends primarily on the distribution of particle sizes in the sample and can be calculated from Stokes' law. A settling time of 5 minutes is usually sufficient for all but the finest-sized samples. The plate, now being loaded with approximately 0.007 mg. of sample per square millimeter, is carefully removed from the petri dish with tweezers and placed to dry, emulsion side up, on a flat surface. When the plate has dried, it should be stored in a light-tight box for exposure. If prolonged exposure (1,000 hours or more) is necessary, surface reactions and fermentation of the gelatin may be minimized by placing the box containing the plate in a refrigerator (Picciotto, 1949, p. 77).

Exposure.—The length of exposure is related to the following: the uranium or thorium content of the sample, the degree to which the uranium or thorium is in radioactive equilibrium with its daughter products, the size and shape of the grains, and the manner in which the radioactivity is distributed in the sample. Generally, a minimum of two diverging alpha tracks is necessary to establish the radioactivity of a grain. Small pitchblende grains can be recognized easily after a 72-hour exposure, but the extremely fine grained radioactive segregations in phosphate rocks require an exposure of at least 1,000 hours. If the length of exposure required for satisfactory results with a mineral grain is unknown, several plates should be prepared at the same time and exposed for different periods of time. Yagoda (1949, p. 177) has calculated that "a fragment measuring 10 microns in average diameter, containing about 50 per cent uranium, will record about 20 tracks per day of exposure. The isolated grain contains less than 10^{-8} g. of uranium . . . ."

Development.—After exposure the plate is developed from 2 to 4 minutes at 68° F. in full strength Eastman Kodak *D-19* developer (or Ilford, *ID*-19). This type of development shows the alpha particle energy-loss rate by changes in the grain spacing of the track. However, if only track counts are to be made, the plate should be developed for 2 minutes at 68° F. in Eastman Kodak *D-8* developer diluted two to one (Webb, 1948, p. 519). Alternate methods of development are given by Yagoda (1949, p. 60). The plate is then rinsed for 10 minutes with two changes of water. The plate is fixed in Eastman Kodak *F-5* fixer or 30 per cent hypo for twice the time required to clear the emulsion and rinsed for 15 minutes with three changes of water. Finally, the plate is allowed to drain and dry thoroughly in a dust-free atmosphere.

Plate covering.—The dispersed-grain methods of Tyler and Marais and others do not include covering the alpha plates. We have found, however, that the addition of a cover glass serves two purposes. First, the alpha plate becomes a permanent mount and may be used repeatedly without danger of dislodging the grains. Second, as most microscope objectives are designed to be used with covered specimens and as alpha plates generally are studied under high-power magnification, the use of a cover glass results in a marked improvement in the resolution of the tracks and mineral grains.

A clean glass cover slip, 22 mm. square, is mounted on the alpha plate with two drops of Canada balsam in xylol. The plate is now kept in an oven in a horizontal position for 48 hours at a temperature of approximately 140° F. After removal from the oven, the plate is allowed to cool before it is examined or stored.

Microscopic examination.—The alpha tracks emitted by the radioactive grains are studied by focusing the microscope down into the emulsion (Fig. 2). Although some alpha tracks may be seen at magnifications of approximately 100 diameters, quantitative track counts cannot be made successfully at magnifications of much less than 500 diameters. Detailed studies of track length and grain spacing should be made at magnifications in excess of 1,000 diameters.

For rapid surveys of the alpha plates a binocular metallographic microscope is used. These surveys are made with transmitted light, a 40× objective, and 15× planar oculars. A 95× oil-immersion objective (*N.A.* = 1.25) is used with the same oculars for detailed track and mineral studies. A larger field can be obtained by using a Bausch and Lomb 40× fluorite oil-immersion objective (*N.A.* = 1.00). For identification of the radioactive minerals a petrographic microscope is employed.

If track-count studies are to be made on previously identified mineral grains, eyestrain can be reduced by the use of a green monochromatic filter or by dark-field illumination.

Advantages and limitations.—The dispersed-grain method may be used to recognize the radioactivity of minerals in a powdered rock sample and may aid in the identification of the minerals. If alpha plates are made under controlled conditions, departures from radioactive equilibrium and the approximate uranium and thorium content may be estimated. A quantitative determination of the uranium content of radioactive minerals by an analysis of the tracks is laborious and time consuming, but a qualitative estimate of the radioactivity of minerals may be made much more easily. Radiocolloids, the localized concentrations of short-lived daughter products (Yagoda, 1946*b*, pp. 462–470), may be studied easily. Alpha plates also may aid in the selection of samples for lead-uranium-ratio age determinations.

This method is limited to minerals that are not water soluble and that are not attacked by photographic solutions. Also, this method can give little information on the spatial distribution of the radioactive minerals in the rock.

Stripping-Film Method

Type of film.—Kodak autoradiographic stripping film consists of a 150-micron supporting film to which has been bonded a 10-micron cellulose ester film covered with a 10- or 25-micron nuclear emulsion sensitive to alpha and beta particles (*NTB*). (See Fig. 3*A*.) These *NTB* stripping films may be obtained from the Eastman Kodak Co., of Rochester,

N. Y. Stripping film is at present available only in boxes containing ten 4-by-5 inch sheets. Approximately 60 days should be allowed for delivery of this film.

Film preparation.—*NTB* emulsions are sensitive to light and should be handled before development under a 25-watt safelight equipped with a Wratten series 2 filter. A sheet of stripping film, measuring 4 by 5 inches, is cut with a print trimmer into eight pieces, each 1 by $2\frac{1}{2}$ inches.

Two score marks, parallel to the length and one-eighth of an inch from each edge, are made through the emulsion and cellulose ester film with a razor blade and straightedge. (See Fig. 3*B*.) As the film is pressure sensitive, care must be taken to avoid touching the part of the emulsion between the score marks. The razor should not cut through the 150-micron supporting base. We have found that the tendency of the film to tear during the stripping process is minimized if the film has been scored as described above.

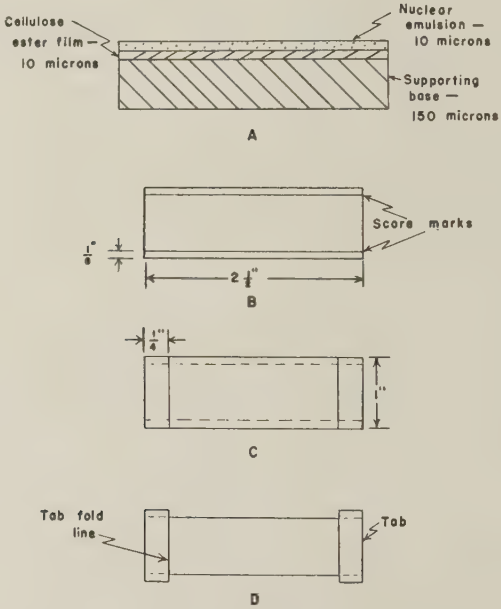
Cellulose ester film — 10 microns
Nuclear emulsion — 10 microns
Supporting base — 150 microns
A
Score marks
1/8"
2 1/2"
B
1/4"
1"
C
Tab fold line
Tab
D

FIG. 3. Nuclear-track stripping film across section and preparation. (*A*) Cross section. (*B*) Film, emulsion side up. (*C*) Film, supporting base up. (*D*) Unsupported emulsion.

Approximately half an inch of one end of the film then is placed, emulsion side down, on a small wooden block and a score 1 inch long and approximately a quarter of an inch from the end of the piece of film is made with a razor blade and straightedge (Fig. 3*C*). This score is deeper than the $2\frac{1}{2}$-inch score marks and should go about halfway through the supporting base. A similar score is made on the other end of the film.

The film, removed from the wooden block, is held by the edges, emulsion side down, and the supporting base is broken carefully along the 1-inch score by bending the tab toward the emulsion. This operation is repeated for the other end of the film. With the film held in the left hand and the tab in the right hand, gently start to strip the emulsion and the cellulose ester film from the supporting base. The emulsion and cellulose ester film should be freed from the supporting base all along the 1-inch score along one end of the film before this operation is repeated for the other end. Now, holding the film with the left hand, emulsion side toward the operator, slowly pull the tab, freeing the emulsion and cellulose ester film from the supporting base. If the film begins to tear, the tear usually will not cross the lengthwise score marks. If the film is stripped without difficulty, the $\frac{1}{8}$-inch ribbons can be removed with tweezers. (See Fig. 3*D*.) It is essential that the film be stripped slowly to prevent fogging by static electricity. It has been found that the film can be stripped more easily on days of high relative humidity. When the relative humidity is low, the film should be placed in a box with a piece of moistened blotting paper for about 15 minutes before the stripping operation is started.

Stripping-film jig.—The jig used to hold the stripping film during mounting is shown in Fig. 4*A*. This jig may be made of metal, wood, or glass slides; its dimensions depend on the size of the specimen to be covered. A rubber band, not shown in Fig. 4, holds the two tab clamps firmly closed. Tweezers are used to place the tabs in the tab clamps of the jig. The loop of film is mounted in the jig with the cellulose ester side up. The loop is centered in the jig and, if necessary, adjusted by means of the tabs until the reflection of the safelight across the top of the loop is parallel to the ends of film jig slide supports. In this position the stripping film is ready to be mounted on the thin section (Fig. 4*B*).

Preparation of thin sections.—Thin sections may be prepared in the usual manner but with the cover glass omitted; however, special precautions against contamination must be taken with samples containing small amounts of uranium. The use of disposable "metallographic" emery papers to minimize contamination has been described by Yagoda (1949, p. 39). The rock slice may be mounted on the glass slide with either canada balsam or Lakeside 70 cement.

In order to allow the thin section of the sample to return to its original radioactive-equilibrium condition and to minimize the loss of daughter products by emanation from hydrous minerals, the thin section should be stored for approximately a month in a desiccator.

Covering thin section.—The uncovered thin section is first dipped in a solution of gelatin at approximately 150° F. This gelatin solution is prepared by placing 12 g. of gelatin (Eastman Kodak flake gelatin, item no. *P1099*) in 150 ml. of distilled water, allowing the gelatin to hydrate for 30 minutes, adding 2.5 ml. of 2 per cent chrome alum and 5 ml. of 0.2 per cent aqueous wetting agent (Yagoda, 1949, p. 95) such as aerosol. The solution then should be gently heated. The gelatin is kept in a tightly stoppered wide-mouth bottle on a hot plate except when the slide is being immersed. When not in use, the gelatin solution is stored in a refrigerator to inhibit bacterial growth. To minimize contamination, very low grade samples should not be dipped in the same gelatin solution that is used for high-grade samples.

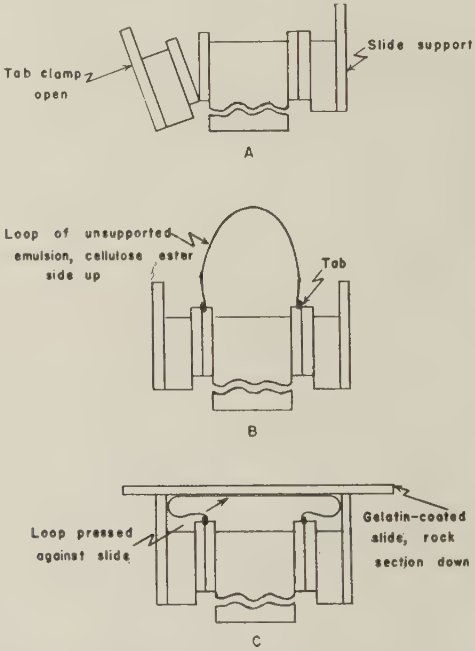
Tab clamp open
Slide support
A
Loop of unsupported emulsion, cellulose ester side up
Tab
B
Loop pressed against slide
Gelatin-coated slide, rock section down
C

FIG. 4. Jig for mounting nuclear-track stripping film. (*A*) Film jig. (*B*) Film mounted in jig. (*C*) Slide resting on slide supports.

After dipping, the slide is allowed to drain for approximately a minute to remove excess gelatin. It is ready to be covered when clear finger-print impressions can be made in the gelatin film on the back of the slide. The slide, rock section down, is centered over the loop of film and gently lowered until the slide rests on the film jig slide supports. (See Fig. 4*C*.)

The slide is chilled by placing a small ice-filled bottle or chilled metal bar on the slide. This rapidly sets the gelatin and prevents the film from pulling away from the thin section during the next operation. The film is cut with a scalpel along the tab fold lines, freeing the film loop from the tabs. After the ice-filled bottle is removed, the slide is taken from the jig. It is placed, thin section up, on the table, and the excess film is removed with a razor and straightedge. If for any reason the slide has been covered imperfectly, the stripping film may be removed by soaking the slide in warm water. A solution of Eastman Kodak rapid mounting cement diluted one to three with ethyl acetate is used to paint down and secure the edges of the film to the slide. The slide is then stored in a light-tight box for exposure.

If the operator is not familiar with stripping film, it is suggested that he make several practice mounts in daylight before attempting this method under darkroom conditions.

Exposure.—The same general factors governing exposure discussed in the dispersed-grain method apply to the stripping-film method. It should be mentioned, however, that, because of the relatively small amounts of material represented by a thin section of a radioactive grain, the exposure time may be increased by a factor of three or four over the exposure time used for the dispersed-grain method. Alpha counts of a uniform, randomly chosen area of the thin section have been made to provide a rough estimate of the exposure time. The optimum exposure time of any particular type of material can best be determined, however, by the exposure of several slides of the same material. If for any reason the exposure was incorrect and another thin section cannot be obtained, the stripping film may be removed from the thin section. This is accomplished by first gently rubbing the cemented edges with a cotton swab dipped in ethyl acetate and then placing the slide in warm (110° F.) water for about 10 minutes.

Development.—The development recommended above for nuclear-track plates may also be used for *NTB* stripping films. Movement of the stripping film due to shrinkage of the emulsion during development and fixation has not been noticed.

Microscopic examination.—In contrast with the dispersed-grain method, the alpha tracks emitted by radioactive grains are studied by focusing up into the emulsion. (See Fig. 5*A* and *B*.) The combination of objectives and oculars used on alpha plates may be used with equal success on stripping film. For oil-immersion work a drop of immersion oil is placed directly on the developed emulsion. The oil is removed by wiping the slide gently with a soft absorbent cloth or tissue. To remove the last traces of the oil, the emulsion is cleaned with a drop of xylol on an absorbent tissue. Because the cellulose ester is isotropic, the determination of the optical properties of the mineral grains in the thin section is made without difficulty.

The use of a vertical illuminator with a metallographic microscope has aided the study of alpha tracks that have been emitted directly over opaque grains. The peripheral tracks emitted by opaque grains can be studied easily by transmitted light.

Advantages and limitations.—The stripping-film method represents an improvement over the mechanical methods of registering autoradiographic plates and thin sections (Poole and Bremner, 1949, p. 130), the method of correlating track patterns with grain shape (Coppens, 1950, p. 21), and the method of simultaneously exposing to light both the alpha plate and the thin section (Picciotto, 1949, p. 79). The use of stripping film assures a positive registration between the developed film and the radioactive grains. Studies have been made that suggest that the cellulose ester film protects the thin section from chemical attack by either the developer or the hypo and minimizes the loss of daughter products by emanation. It should be mentioned that great dexterity is not required to cover thin sections with nuclear-track stripping film. After several trials in daylight the average operator can make satisfactory mounts under darkroom conditions.

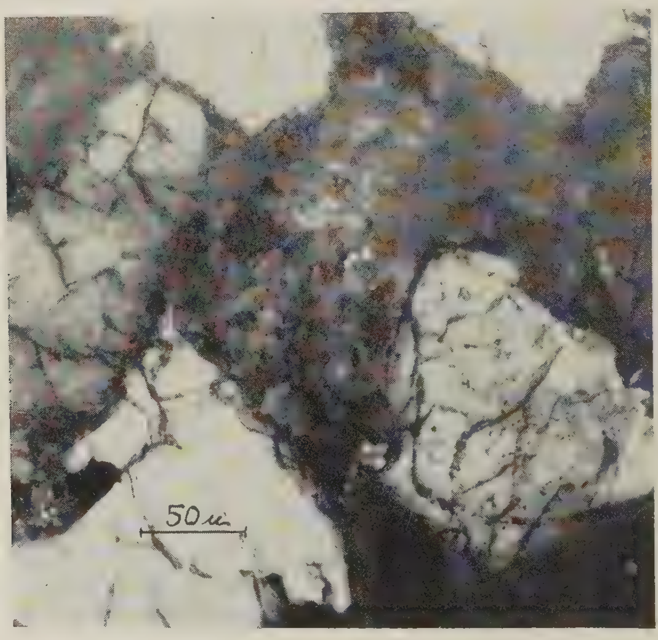

FIG. 5*A*. A photomicrograph of carnotite-bearing sandstone from the Rock Creek area, Montrose County, Colo. The white and gray fractured grains are quartz and the interstitial areas contain microcrystalline carnotite. Crossed nicols.

Some of the inherent limitations of the stripping-film method are a consequence of the methods of preparing the thin sections. Radioactive minerals may be lost easily in grinding; and, if precautions are not taken, the slide may be contaminated by the re-use of grinding powders and rouges. A thorough study of the distribution of radioactivity in a rock can be made only by examining many thin sections. Approximately a week's exposure is required before any results can be obtained. The stripping-film method is also limited by the activity of the material under investigation. Generally, two alpha tracks are necessary to locate a radioactive grain because the alpha tracks must be projected through the 10-micron cellulose ester film. Also quantitative track length studies are not possible because only part of the total track is recorded in the 10-micron emulsion and some alpha particle energy is lost in traversing the 10-micron cellulose ester film.

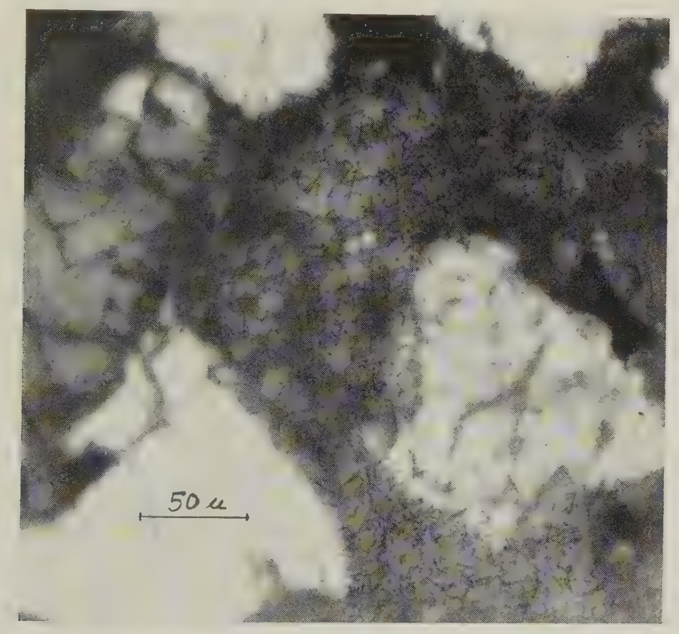

Fig. 5*B*. A photomicrograph of the alpha tracks overlying the interstitial carnotite in the field shown in figure 5*A*. The exposure time for the nuclear-track stripping film was 6 days. Crossed nicols.

Recently Ford (1951, p. 273) has described the use of liquid nuclear emulsion to study radioactive minerals in thin section. This high-resolution method should be ideal when rocks and minerals of very low radioactivity are being examined, as the alpha tracks can be followed directly to their source.

We believe that the stripping-film method, described above, may be used without modification for histological studies and with some modification for polished-section and metallographic studies.

Acknowledgments

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SPHERULITIC BRECCIAS IN A DOME NEAR WENATCHEE, WASHINGTON

Howard A. Coombs, *University of Washington, Seattle, Washington.*

Abstract

A felsitic dome located on an anticlinal axis in the lower Eocene Swauk arkose in central Washington has partial borders of perlite and felsitic breccia. Portions of the perlite and the breccia have been converted into a mass of spherulites comprising up to 80 per cent of the volume of the rock. Chemical analyses of the spherulitic breccia, the perlite and the arkoses give clear evidence as to what has been added metasomatically to cause the transformation to spherulites. The arkose and the perlite are remarkably similar in chemical composition. The analysis of the spherulites shows that potash and silica have been added, probably accompanied by a release of water from the perlite during the course of crystallization. Brecciated margins of the dome have provided avenues of transfer for the solutions and it is in these zones that the spherulites have had their most abundant growth.

Introduction

Wenatchee is located on the west bank of the Columbia River in the geographic center of the state of Washington. The rocks in this vicinity are a series of arkoses, shales, and conglomerates, known as the lower Eocene Swauk formation, folded into a series of northwest-southeast trending anticlines and synclines. The Yakima basalts of Miocene age overlie the truncated folds of the Swauk sediments with a marked unconformity and they also form the vast Columbia Plateau on the east side of the Columbia River.

On the southern city limits of Wenatchee a dome rises 150 feet above the orchard covered terraces and forms a distinctive landmark. This dome was intruded along the axis of one of the major anticlines in the Swauk arkoses. It is the purpose of this paper to describe the rock types in this dome, to explain the origin of the rocks now converted almost wholly to spherulites and to give some evidence as to the nature of the metasomatic solutions that took part in this transformation.

As early as 1904 Smith and Calkins noticed the "Pronounced lamination more or less parallel to the surface on all parts of the dome." In 1936 Chappell mapped the Wenatchee quadrangle and again mentioned the striking jointing forming plates parallel to the periphery. He also called attention to the perlite exposed on the southeast side and to the friable sand resulting from the disintegration of portions of the perlite. As a result of Chappell's careful work the thickness of the perlite was established as approximately 50 feet, rather wide for a chilled selvage on a dome 400 feet in diameter.

The wide interest in perlite as an industrial material during the past few years has focused attention on all perlite deposits. Huntting (1949) reviewed the deposits within this state and made an investigation of the Wenatchee locality. He described a thin section from Wenatchee showing 80 per cent glass and the remainder composed of crushed quartz and plagioclase phenocrysts with a few scattered crystals of biotite and hornblende. A partial analysis of the rock and the expansion qualities were given by Huntting.

OCCURRENCE

This dome forms but one of many intrusives located along the axis of an anticline in the Swauk arkoses and shales of lower Eocene age. In common with the major structural trends in central and western Washington this axis strikes in a general northwest-southeast direction. The intrusives vary greatly in composition along the axes of the folds in the sediments. Basalts, andesites, dacites, rhyolites and their porphyries are all represented. The more basic varieties are usually in the form of sills, dikes, or laccoliths. The more acid types are roughly circular or irregular in shape and many have been altered intensely by hydrothermal solutions.

The most striking feature of the dome is the concentric platy jointing. Sheets several feet in diameter may have a thickness of one-half inch or less. The parallelism of minerals and the distinct bands of slightly different color indicate flow banding and the platy parting is obviously parallel to the flow-banding. The sheets dip away from a common center with angles varying from 25 to 55 degrees.

The core of the dome is a white porphyritic (or xenocrystic) felsite. Along the southern margin is a rim of medium gray banded perlite approximately 50 feet thick. The northern border of the dome is rimmed by a breccia composed of gray felsitic blocks in a matrix of similar material but darker gray. Locally hydrothermal alteration is very marked. Spherulites developed in this breccia and in much of the core contiguous to the breccia. In places over 80 per cent of the rock is composed of spherulites.

PETROGRAPHY AND PETROLOGY

The core is composed of a white, platy, porphyritic (or xenocrystic) felsite with crystals or pieces of quartz averaging one millimeter in diameter making up to 15 per cent of the volume of the rock. Less abundant than the quartz is plagioclase (An27) exhibiting strong zoning with more sodic rims or, if the zoning is lacking, they may be deeply embayed or almost completely replaced with spherulites. The sodic rims seemed to have served as a protection against reaction with the magma and only those with rims now have euhedral shapes.

Brown biotite and pale green hornblende are present in very small

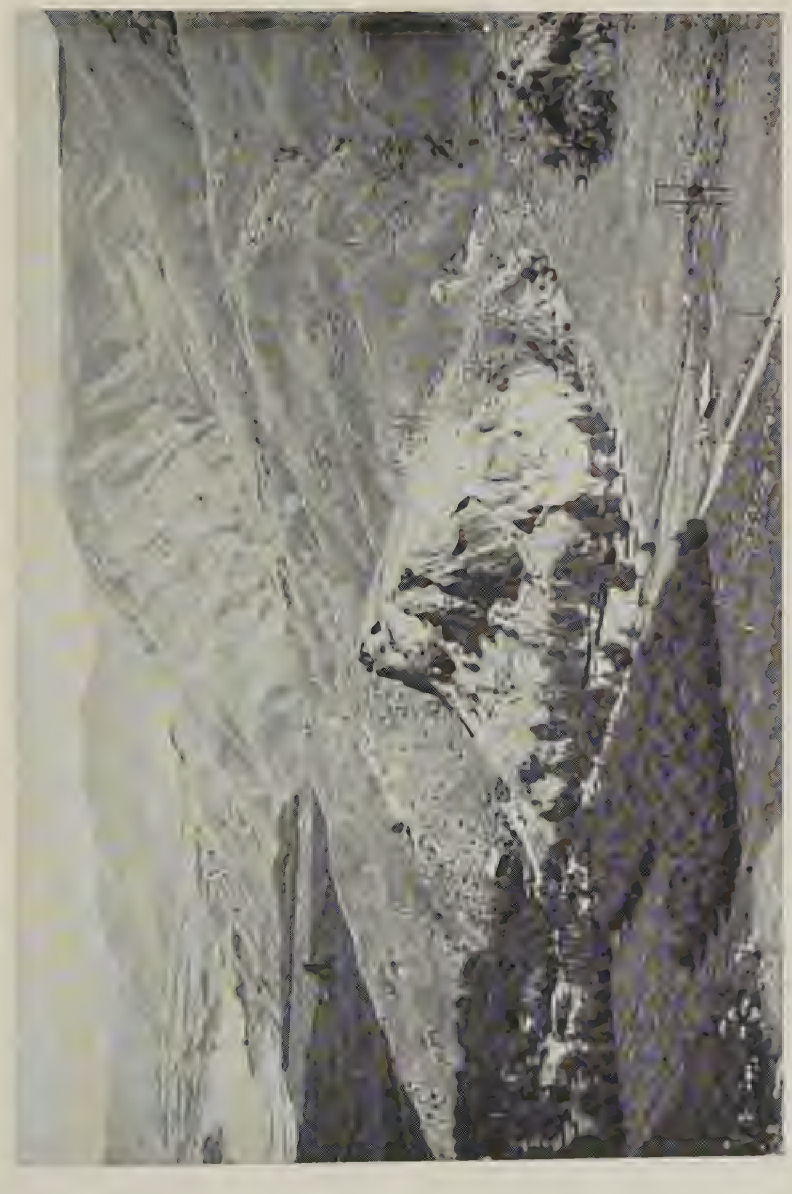

FIG. 1. The Wenatchee dome looking southeast along the anticlinal axis in the Swauk. The Columbia River is visible in the upper left. The low ridge on the right of the dome is the silicified arkose, the low ridge on the left is the quartzitic conglomerate. The perlite is partially exposed on the right side of the dome.

quantities. The quartz is peculiar in that the shapes may be sharp and jagged or they may be rounded and very deeply embayed. Such shapes are characteristic in the neighboring arkose and silicified conglomerate. Even more suggestive of the incorporation of the arkose fragments in the dome rocks is the distinctive undulatory extinction so typical of the rounded quartzite pebbles and the angular clastic grains of the arkose. Most of the quartz and some of the feldspars thus appear to be xenocrysts instead of phenocrysts. Ross (1948) found similar fragments of quartz and feldspar in a fused glass in New Mexico.

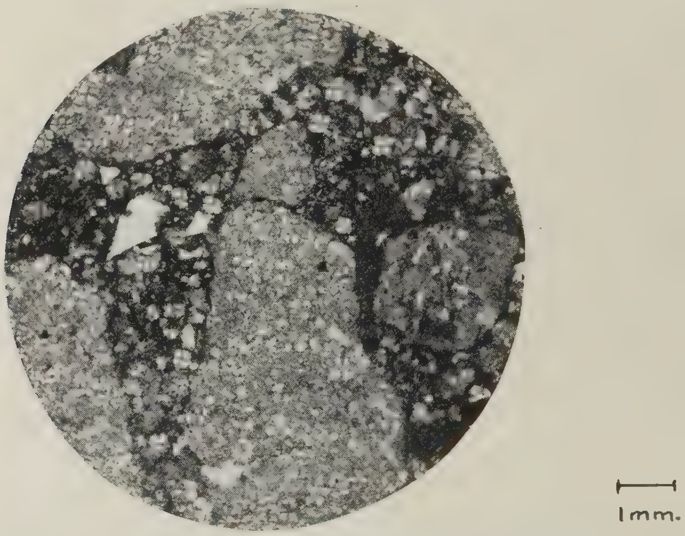

FIG. 2. Spherulitic breccia under crossed nicols. The small black crosses indicate the presence of spherulites both in the blocks and in the matrix.

The perlite where best exposed on the southern margin of the dome is a medium gray rock with alternating bands of light and dark gray material. The crystals are approximately the same size as those in the core rock but more xenocrystic. In some sections the quartz is strung out in a line of angular fragments parallel to the flow banding as in a protoclastic border. In the perlite the plagioclase is either angular or deeply embayed but does not show the addition of more sodic rims. The glass in the perlite has an index of 1.492 indicating a high silica content (73%) according to the tables by George (1924) and this is verified by the chemical analysis. Clear belonites are plentiful in the perlite and pass across the concentric cracks without interruption. Most belonites show a subparallel arrangement undoubtedly due to flowage.

The breccias are composed of light gray blocks up to several inches in diameter set in a darker gray matrix of finely comminuted fragments (see Fig. 2). The breccia is firm and brittle where late hydrothermal action has not been severe. The lighter colored blocks resemble the perlite or central core felsite under the microscope in that they are clear and charged with belonites oriented in a sub-parallel fashion. The perlitic cracks, however, are lacking. Under crossed nicols the development of spherulites is striking for they make up from 60 to 80 per cent of the rock and occur in the darker matrix as well as in the blocks.

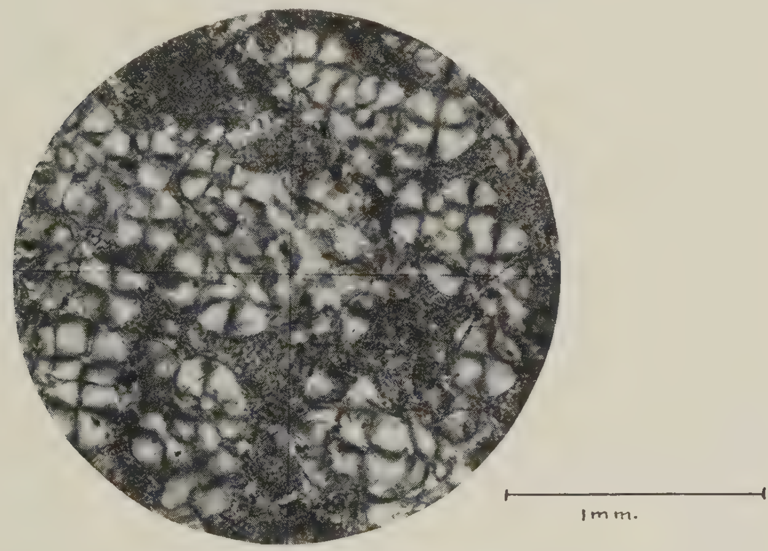

FIG. 3. Spherulites in the blocks under higher magnification and crossed nicols.

The spherulites are uniform in optical properties and each spherulite is homogeneous throughout its mass (see Fig. 3). Indices determined were $Nz=1.530$ and $Ny=1.528$. These together with the other optical properties suggests a perthitic composition for the spherulites. In his x-ray determination of sub-microscopic spherulites Hurlbut (1936) inferred that they contained perthite and cristobalite. The spherulites from Wenatchee may be very similar but quartz is present in many of the cavities. Tiny spherulites may occur between some of the larger ones but a remarkable uniformity in size exists in that most of the larger spherulites attain a diameter of 0.2 mm. In the matrix where iron oxides are more prevalent the spherulites have concentrated the iron oxides in the center of each spherulite leaving a clear, rounded rim.

The belonites still retain their sub-parallel alignment in the spherulites within the blocks in the breccia. Perhaps this is due in part to the size of the belonites for they may be as long as the diameter of the spherulite, or one belonite may lie across the boundaries and within two spherulites. This indicates the spherulites were formed after all movement had ceased and that all formed at approximately the same time. Similar evidence was used in deciphering the origin of the Yellowstone vitrophyres by Howard and Colony (1934 and 1939).

Perlite and Spherulitic Breccia. In a search through Professional Paper No. 99 (1917) for analyses of perlites, pitchstones, and obsidians all 80 examined showed a decidedly higher percentage of potash than is found in the Wenatchee perlite. Those with a relatively low potash content were also very much lower in silica. Even the soda-rhyolite of Yellowstone (Iddings, 1885) had twice as much potash as occurs in the glassy phase of the dome at Wenatchee. Most all of the obsidians were far more deficient in water. The pitchstones and perlites averaged between 3 and 4 per cent of water but the extremes were between one and 13 per cent. Thus the Wenatchee perlite is most unusual in its extremely low potash content. Less unusual is the moderately high water content.

TABLE OF ANALYSES

	1	2	3	4
SiO_2	73.33	74.11	77.30	89.95
Al_2O_3	15.27	11.20	12.39	4.94
Fe_2O_3	3.25	.52	.57	.43
FeO	.11	.28	.19	.18
MgO	.15	.23	.10	.04
CaO	.09	1.88	.68	.14
Na_2O	.09	3.17	3.14	.34
K_2O	1.38	1.04	4.36	3.10
H_2O+	5.30	5.14	.60	.43
H_2O-	.35	2.20	.31	.11
TiO_2	.70	.11	.12	.07
P_2O_5	.07	.02	.02	.02

No. 1 Arkose, Eileen H. Kane, analyst.
No. 2 Perlite, Eileen H. Kane, analyst.
No. 3 Spherulitic breccia, Eileen H. Kane, analyst.
No. 4 Silicified arkose, Eileen H. Kane, analyst.

A glance at the table of analyses shows a striking difference between the potash content of the perlite and the spherulitic breccia. Since the breccia is composed predominantly of spherulites it may be assumed that the analysis for this rock corresponds very closely to the analysis of the spherulites themselves. The chemical analysis of the spherulitic breccia is almost identical with that of the small dark blue spherulites from

Obsidian Cliff in Yellowstone National Park. If the spherulitic breccia is formed at the expense of the perlite, or possibly the glassy to cryptocrystalline material of the core, then the greatest change involved is the addition of potash to form the spherulites and the loss of water as crystallization progressed. A decrease in CaO and an increase in silica is also noteworthy.

All evidence points to the formation of the spherulites from the glassy to cryptocrystalline material of the core, or from the clear perlites. The belonites so typical of the glasses are still present in the spherulites and with approximately the same orientation. Many sections show portions of the glass partially converted to spherulites. The chemical analyses are so similar (except for the potash and water) that no doubt exists as to their common origin. A separate intrusion may be postulated for the spherulitic material but the intimate admixture with the glass precludes this possibility. All other studies in the literature of spherulites point to their growth at the expense of the enclosing glass.

The conditions of spherulitic growth have been well described by Foshag (1926).

> The solubility of silica in gaseous water may be fairly great at comparatively low temperatures. The solubility need not be great, nor the amount of solvent large for the solvent in a case such as this acts as a catalyzer. Its action is continuous. As rapidly as the crystalline phase separates from its super-saturated solution the solvent is capable of dissolving more glass, the process continuing until the transformation is complete or until the escape of solvent stops the reaction. In this manner the final effect of a small amount of solvent can well be the transformation of a considerable bulk of glass into crystalline matter.

At Wenatchee the effective solvent was potash and water, probably in the gaseous form. Locally the transformation to spherulites has been almost complete.

The brecciation so characteristic of domes, as pointed out by Williams (1931), provided a porous zone for hydrothermal solutions. As a result the strongest alteration hydrothermally and the most complete transformation to a mass of spherulites is in, or contiguous to, the zone of brecciation.

Arkoses. In dealing with instrusive igneous masses it is well to consider the country rock and its structure. Two ridges of Swauk sediments occur on either side of the dome (see Fig. 1). The one 150 feet to the east is a conglomerate composed essentially of rounded quartzite pebbles ranging from one to six inches in diameter. The larger pebbles are rounded but in thin section the smaller fragments of quartz are very angular. Most all of the quartz shows a wavy extinction. Crumpled flakes of leached biotite and muscovite are also present. This bed strikes N 50° W and dips 60° to the N.E. The ridge 200 feet to the west of the dome is a very silicified arkose striking parallel to the other ridge but dipping steeply to the southwest. The dips are not primarily due to local doming for an anticlinal axis extending for several miles passes through the dome and elsewhere along this axis the dips are steep. One would expect the ridges on either side to be bowed outward around the dome. The exposures of the ridges adjacent to the dome are not continuous and hence it is difficult to determine if bowing has occurred or not. The meagre exposures indicate that they are not definitely bowed out and certainly not sufficient to allow the emplacement of so large a dome.

Two samples of the Swauk arkose were selected for analysis. Sample No. 1 was a half mile from the dome and showed no indication of igneous or hydrothermal action. This is a buff colored arkose, quite firmly cemented but it can be broken with the fingers along the weathered outcrop. Sample No. 4 is from the ridge of silicified arkose immediately west of the dome. These samples were selected in the hope that they would give some clue as to what influence the country rock might have had on the instrusive and also what might have escaped from the intrusive into the country rock.

Most unusual is the striking similarity of the arkose some distance from the dome (No. 1) and the perlite (No. 2). The percentages of silica, potash and water are extremely close although the localities are separated by more than half a mile and one rock is distinctly sedimentary—the other definitely igneous. The higher alumina and ferric oxide content of the sediment is to be expected on a weathered surface even if the rocks originally had the same composition. One may well wonder how close the arkose would be to the perlite if fused.

Chemically they are very similar and xenocrysts of the sediment are scattered in the glass and in the core rock. In this connection it is noteworthy that 2½ miles southeast of the dome a well was drilled on the axis of an anticline in the Swauk in the search for oil. At a depth of 821 feet below the surface the well penetrated 8 feet of perlite and 169 feet of perlite in the lower part of a 256 foot thick rhyolite body (Huntting, 1949). Of course it is unlikely that these are true thicknesses for the penetration of a drill through a body of unknown orientation could give a true thickness in only one position.

Relation of Perlite to Felsite. The relation of the perlite to the felsite is somewhat obscure. The perlites may represent chilled borders but the thicknesses are unusually large for the size of the intrusives. It is very doubtful if a perlitic border extends more than half way around the dome. They may represent separate intrusions of perlite and felsite. However, even in the well the perlite was encountered at four separate horizons in the rhyolite. The gradations between the perlite and felsite at the dome, and their striking similarity in chemical composition would be evidence against separate intrusions. As pointed out by Higazy (1950) two such rock types could exist together as differentiates from a soda rich and a potash rich magma, but not as differentiates from a common basaltic magma. As he also points out they could be formed by metasomatic processes if the field and textural studies so indicated. The field and chemical evidence at Wenatchee gives every indication that metasomatism has been operative in the vicinity of the dome and elsewhere.

These masses may be due to local fusion of the arkoses either by hot alkaline solutions or by the intrusion of the rhyolitic magma or both. The lack of needed displacement in the ridges adjacent to the dome lends support to this idea. The striking similarity in chemical composition of the perlite and the arkose, together with the fact that the perlite has a most unusual composition, is indicative of this origin. The presence of xenocrysts of the arkose in the perlite in various states of being resorbed is suggestive of some fusion or reaction. However, the evidence for the origin of the perlites is not conclusive at Wenatchee.

Origin of the Spherulitic Breccias. It is quite evident that the spherulites were formed from the perlite or glassy portions of the felsite aided by metasomatism. The silicified arkoses adjacent to the dome give some indication that silica has been added. Silica was used in forming the spherulites so none was released by the dome rocks. Silica, therefore, was probably added metasomatically and many other localities near the dome show excellent evidence of silica and alkali metasomatism (Coombs, 1950).

The potash content of the silicified arkose has increased greatly in proportion to the other oxides. All values are depressed because of the very high silica content. Potash was also used in forming the spherulites so no excess was available from the perlites. This too must have been added metasomatically. Terzaghi (1948) described and gave evidence for a very similar type of potash and silica metasomatism in the obsidians at the Esterel, France.

Water was released from the perlite during its crystallization and in all probability it too was added metasomatically. Its avenues of transfer are marked by hydrothermal alteration both within the dome and within the sediments.

Thus the spherulites at Wenatchee seem to have been formed at the expense of the glass but the process was aided considerably by potash and silica metasomatism. Water, probably in the gaseous form, may have been added but much was released during the course of crystallization of the perlite.

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APPROXIMATE FORMULAE FOR TRICLINIC CALCULATIONS*

A. L. PATTERSON, *The Institute for Cancer Research and The Lankenau Hospital Research Institute, Philadelphia 11, Pennsylvania.*

ABSTRACT

Approximation formulae are presented which allow the calculation of the elements of a triclinic lattice from those of the corresponding reciprocal lattice and vice-versa. These formulae, which do not require the use of trigonometric tables, are highly accurate for lattice angles close to 90°. Their value for approximate calculations when the angles depart from 90° is also discussed in the paper.

The usual formulae for the calculation of the elements of the triclinic system (cf. Buerger 1942, Chap. 18) are cumbersome and require many references to trigonometric tables. Various graphical and nomographic methods have been suggested (e.g. Bond 1950) but all these require special facilities. The present note presents a type of approximation formula which is surprisingly accurate, is well adapted to machine or slide rule calculation, and avoids the use of trigonometric tables.

Consider first the formula

$$\cos\alpha = \frac{\cos\beta^*\cos\gamma^* - \cos\alpha^*}{\sin\beta^*\sin\gamma^*} \tag{1}$$

which with its inversion and with cyclic permutations permits the calculation of the crystal lattice angles α, β, γ from the reciprocal lattice angles $\alpha^*\beta^*\gamma^*$ and vice versa. Adopting the notation

$$\bar{\alpha} = \pi/2 - \alpha \text{ (etc.)}, \tag{2}$$

we may write (1), in terms of the complements of the angles, as

$$\sin\bar{\alpha} = \frac{\sin\bar{\beta}^*\sin\bar{\gamma}^* - \sin\bar{\alpha}^*}{\cos\bar{\beta}^*\cos\bar{\gamma}^*}\,. \tag{3}$$

Using standard methods, discussed briefly below, this expression may be expanded in series in the form

$$\bar{\alpha} \approx -\bar{\alpha}^* + \bar{\beta}^*\bar{\gamma}^* - \tfrac{1}{2}\bar{\alpha}^*(\bar{\beta}^{*2} + \bar{\gamma}^{*2}) \tag{4}$$

where the angles are expressed in radians. If the angles are expressed in degrees, (4) must be written

$$\begin{aligned}\bar{\alpha} &\approx -\bar{\alpha}^* + \mathrm{K}\bar{\beta}^*\bar{\gamma}^* - \tfrac{1}{2}\mathrm{K}^2\bar{\alpha}^*(\bar{\beta}^{*2} + \bar{\gamma}^{*2})\\ &= -\mathrm{T}_1 + \mathrm{KT}_2 - \mathrm{K}^2\mathrm{T}_3.\end{aligned} \tag{5}$$

The two fourth degree terms omitted from the expansion (5) are

$$\mathrm{K}^3(\mathrm{T}_4 + \mathrm{T}_4') = \tfrac{1}{2}\mathrm{K}^3\bar{\alpha}^{*2}\bar{\beta}^*\bar{\gamma}^* + \tfrac{1}{3}\mathrm{K}^3\bar{\beta}^*\bar{\gamma}^*(\bar{\beta}^{*2} + \bar{\gamma}^{*2}). \tag{5'}$$

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In these expressions, the symbols T_1, T_2, etc., are introduced to simplify the later discussion, and the constant $K = \pi/180°$ and its powers have the values:

$$\begin{aligned} K &= 1.7453 \times 10^{-2} & K^{-1} &= 5.7296 \times 10 \\ K^2 &= 3.0462 \times 10^{-4} & K^{-2} &= 3.2828 \times 10^{3} \\ K^3 &= 5.3166 \times 10^{-6} & K^{-3} &= 1.8809 \times 10^{5} \\ K^4 &= 9.2792 \times 10^{-8} & K^{-4} &= 1.0777 \times 10^{7} \end{aligned}$$

In Table 1, the errors (ϵ) for the expansion (5) and its abbreviated forms are calculated for the several ranges $\epsilon = 0.5°$, $0.05°$, $0.005°$ which correspond to calculations which may be taken as valid respectively to 1°, 0.1°, 0.01°. Under each formula and each value of ϵ the useful range is expressed in two ways: in the upper line as the maximum range for the leading term (T_2, T_3, etc.) omitted, and in the lower line in terms of the angle range (AR) permitted in the most unfavorable case in which all the angles involved in the omitted term have the same value. The error ranges chosen for calculation in Table 1 are based on the errors met with in the usual goniometric and x-ray analytic techniques. Most work should be better than $\epsilon = 0.05°$ which corresponds to a value accurate to 0.1°, while it is only exceptional work which reaches $\epsilon = 0.005°$ accurate to 0.01°.

TABLE 1. ERROR ANALYSIS FOR EQUATION (5)
Angles expressed in degrees

Approximation formula	Term omitted	Permitted Error ϵ		
		$\epsilon < 0.5°$	$\epsilon < 0.05°$	$\epsilon < 0.005°$
(5*a*) $\bar{\alpha} = -\bar{\alpha}^*$	T_2	< 28.6	< 2.86	< 0.286
	AR	$< 5.35°$	$< 1.69°$	$< 0.54°$
(5*b*) $\bar{\alpha} = -\bar{\alpha}^* + K\bar{\beta}^*\bar{\gamma}^*$	T_3	$< 1.64 \times 10^3$	$< 1.64 \times 10^2$	$< 1.64 \times 10$
	AR	$< 11.8°$	$< 5.47°$	$< 2.54°$
(5) $\bar{\alpha} = -\bar{\alpha}^* + K\bar{\beta}^*\bar{\gamma}^* - \frac{1}{2}K^2\bar{\alpha}^*(\bar{\beta}^{*2} + \bar{\gamma}^{*2})$	T_4	$< 9.40 \times 10^4$	$< 9.40 \times 10^3$	$< 9.40 \times 10^2$
	AR	$< 20.8°$	$< 11.7°$	$< 6.59°$
	T_4'	$< 9.40 \times 10^2$	$< 9.40 \times 10^3$	$< 9.40 \times 10^2$
	AR	$< 19.4°$	$< 10.9°$	$< 6.13°$

From Table 1, it is clear that the simplest formula

$$\bar{\alpha} = -\bar{\alpha}^* \qquad (5a)$$

is of value only for the roughest calculations. It will however give a determination of the correct sign[1] for $\bar{\alpha}$ in all cases in which $\bar{\alpha}^*$ and $\bar{\beta}^*\bar{\gamma}^*$ have opposite sign, and in those cases of the same sign in which $|\bar{\beta}^*\bar{\gamma}^*| > 57.3|\bar{\alpha}^*|$, i.e. (5*a*) will then give the incorrect sign when $\bar{\alpha}^*$ is very small compared with $\bar{\beta}^*$ and $\bar{\gamma}^*$. The second approximation given by the formula

$$\bar{\alpha} = -\bar{\alpha}^* + K\bar{\beta}^*\bar{\gamma}^* \tag{5b}$$

is adequate for almost all crystallographic work for angles up to 3° or 4° and for rough calculations it can be used up to 6°. The third approximation of (5) will enable the calculations to be carried out to 0.01° for angles up to 6° and will be satisfactory for many calculations up to 10° or 12°. It is of course possible to add the fourth degree terms of equation (5′) to those of (5) if higher precision is desired over a wider range of angle, but under such circumstances it is undoubtedly better to have recourse to the original expression (1) and the trigonometric tables.

If we solve equation (1) for cos α^* we obtain the expression

$$\cos\alpha^* = \cos\beta^*\cos\gamma^* - \sin\beta^*\sin\gamma^*\cos\alpha, \tag{6}$$

which is used in those cases in which one crystal angle and two reciprocal angles are measured (e.g. on the Buerger precession camera when two zero layers at a known angle apart have been photographed). Equation (6) can be expanded in the form

$$\bar{\alpha}^* = -\bar{\alpha} + K\bar{\beta}^*\bar{\gamma}^* + \tfrac{1}{2}K^2\bar{\alpha}(\bar{\beta}^{*2} + \bar{\gamma}^{*2}). \tag{7}$$

Since the terms of this expression differ only in detail from those of (5), the error discussion of Table 1 is directly applicable here.

The expanded form of the expression for the volume of the cell is particularly convenient and quite accurate. The usual expression

$$V = V_0[1 - \cos^2\alpha - \cos^2\beta - \cos^2\gamma + 2\cos\alpha\cos\beta\cos\gamma]^{1/2} \tag{8}$$

becomes

$$V = V_0[1 - \tfrac{1}{2}K^2(\bar{\alpha}^2 + \bar{\beta}^2 + \bar{\gamma}^2) + K^3\overline{\alpha\beta\gamma}], \tag{9}$$

in which $V_0 = abc$. The largest term omitted is

$$K^4T_4 = (K^4/24)[(\bar{\alpha}^4 + \bar{\beta}^4 + \bar{\gamma}^4) - 6(\bar{\beta}^2\bar{\gamma}^2 + \bar{\gamma}^2\bar{\alpha}^2 + \bar{\alpha}^2\bar{\beta}^2)]. \tag{9′}$$

The error analysis for (9) is given in Table 2. From it we see that the simplest result $V = V_0$ is sufficiently accurate for the determination of the number of molecules per cell even for quite large angles. The complete expression (9) which is quite simple to use is adequate for almost all

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[1] Difficulty cannot arise from the term T_3 since it always has the sign of T_1. The terms T_4 and T_4' cannot enter unless the complementary reciprocal lattice angles reach the extraordinary values of 70–80°.

TABLE 2. ERROR ANALYSIS FOR EQUATION (9)
Angles expressed in degrees

Approximation formula	Term omitted	Permitted Error ϵ		
		$\epsilon<5\%$	$\epsilon<0.5\%$	$\epsilon<0.05\%$
(9*a*) $V=V_0$	T_2 AR	<161 $<10.5°$	<16.1 $<3.31°$	<1.61 $<1.05°$
(9*b*) $V=V_0[1-\frac{1}{2}K^2(\bar{\alpha}^2+\bar{\beta}^2+\bar{\gamma}^2)]$	T_3 AR	$<9.40\times10^3$ $<21.1°$	$<9.40\times10^2$ $<9.80°$	$<9.40\times10$ $<4.55°$
(9) $V=V_0[1-\frac{1}{2}K^2(\bar{\alpha}^2+\bar{\beta}^2+\bar{\gamma}^2)+K^3\bar{\alpha}\bar{\beta}\bar{\gamma}]$	T_4 AR	$<5.39\times10^5$ $<30.5°$	$<5.39\times10^4$ $<17.1°$	$<5.39\times10^3$ $<9.64°$

structure analytical calculations provided the cell angles are within 10° of right angles.

It is perhaps worth while to outline briefly the steps involved in one of these expansions. Equation (3) may be rewritten in the form

$$\sin\bar{\alpha} = \tan\bar{\beta}^*\tan\bar{\gamma}^* - \sec\bar{\beta}^*\sec\bar{\gamma}^*\sin\bar{\alpha}^*.$$

The power series for the trigonometric functions (see, for example, Pierce 1929, pp. 91–92) permit the expansion

$$\begin{aligned}\sin\bar{\alpha} = {} & (\bar{\beta}^* + \bar{\beta}^{*3}/3)(\bar{\gamma}^* + \bar{\gamma}^{*3}/3) \\ & - (1 + \bar{\beta}^{*2}/2 + 5\bar{\beta}^{*2}/24)(1 + \bar{\gamma}^{*2}/2 + 5\bar{\beta}^{*2}/24)(\bar{\alpha}^* - \bar{\alpha}^{*3}/6)\end{aligned}$$

in which terms higher than the fourth power in the angles are omitted. Carrying out the multiplication we get

$$\sin\bar{\alpha} = -\bar{\alpha}^* + \bar{\beta}^*\bar{\gamma}^* - \tfrac{1}{2}\bar{\alpha}^*(\bar{\beta}^{*2} + \bar{\gamma}^{*2}) + \bar{\alpha}^{*3}/6 - \tfrac{1}{2}\bar{\alpha}^*(\bar{\beta}^{*2} + \bar{\gamma}^{*2}).$$

We now invert the usual series for sin $\bar{\alpha}$ in the form

$$\bar{\alpha} = \sin\bar{\alpha} + (\sin^3\bar{\alpha})/6$$

and obtain

$$\bar{\alpha} = -\bar{\alpha}^* + \bar{\beta}^*\bar{\gamma}^* - \tfrac{1}{2}\bar{\alpha}^*(\bar{\beta}^{*2} + \bar{\gamma}^{*2}) + \tfrac{1}{2}\bar{\alpha}^{*2}\bar{\beta}^*\bar{\gamma}^* + \tfrac{1}{3}\bar{\beta}^*\bar{\gamma}^*(\bar{\beta}^{*2} + \bar{\gamma}^{*2}).$$

Similar techniques can be applied to the expansion of (6) and (8) and to the discussion of similar expansions of other formulae which may interest the reader.

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QUANTITATIVE DETERMINATION OF CALCITE ASSOCIATED WITH CARBONATE-BEARING APATITES*

Sol R. Silverman, Ruth K. Fuyat, and Jeanne D. Weiser

Abstract

The CO_2 combined as calcite in carbonate-bearing apatites has been distinguished from that combined as carbonate-apatite, or present in some form other than calcite, by use of x-ray powder patterns, differential thermal analyses, and differential solubility tests. These methods were applied to several pure apatite minerals, to one fossil bone, and to a group of phosphorites from the Phosphoria formation of Permian age from Trail Canyon and the Conda mine, Idaho, and the Laketown district, Utah. With the exceptions of pure fluorapatite, pure carbonate-fluorapatite, the fossil bone, and one phosphorite from Trail Canyon, these substances contain varying amounts of calcite, but in all the samples an appreciable part of the carbonate content is not present as calcite. The results of solubility tests, in which the particle size of sample and the length of solution time were varied, imply that the carbonate content is not due to shielded calcite entrapped along an internal network of surfaces.

Introduction

The existence of carbonate-apatite as a distinct mineral constituent of bone, teeth, and phosphorite is not universally accepted. Hendricks and Hill (1950, pp. 731–737) attribute the carbonate content of bone and of all carbonate-bearing apatites to carbonate entrapped in hydroxylapatite or in hydrated tricalcium phosphate. Other workers (Dallemagne, Brasseur, and Mélon, 1949, pp. 138–145; Dallemagne and Mélon, 1945, pp. 597–599) claim that bone is a mixture of tricalcium phosphate and calcium carbonate, but they do not question the existence of carbonate-apatite as a distinct mineral phase in dentine. McConnell and Gruner (1940, pp. 157–167) recognize carbonate-apatite as a single homogeneous mineral. The controversy has been summarized by Geiger (1950, pp. 161–181).

Staining methods (see appendix) have indicated that an appreciable part of the carbonate content of natural phosphorites is present as calcite. In order to determine quantitatively the amounts of carbonate present as calcite and as carbonate not combined as calcite, carbonate-bearing phosphorites were examined by three independent methods: x-ray analysis, differential thermal analysis, and differential solubility tests.

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X-Ray Analysis

Optically homogeneous apatites containing as much as 5.8 per cent carbon dioxide have been reported. Whether the carbon dioxide is present as calcite or as carbonate in some other form, or is integrated in the apatite structure, these substances would be expected to give *x*-ray patterns distinguishable from those of carbonate-free apatites. In 1933 Bredig, Franck, and Füldner (1933, pp. 959–969) reported having recognized distinct differences in the *x*-ray diffraction pattern of a carbonate-hydroxylapatite as compared with those of fluorapatite and hydroxylapatite. The controversy that followed is reflected in the work of Thewlis, Glock, and Murray (1939, pp. 358–363), and McConnell and Gruner (1940, pp. 157–167).

Small but measurable differences in the *x*-ray patterns of carbonate-fluorapatite, hydroxylapatite, and fluorapatite have been detected in the course of this investigation, and a complete account of these differences will be published later. The distinction is based on variations in spacings between the (231) and (004) lines in *x*-ray powder films exposed on a 57.3-mm. radius camera using nickel-filtered copper radiation.

The variations of *x*-ray data listed in Table 1 were obtained from two hydroxylapatites (from Holly Springs, Ga., and a synthetic product prepared by W. L. Hill of the U. S. Department of Agriculture), from two fluorapatites (from Cerro Mercado, Durango, Mexico, and Ontario, Canada), and from two carbonate-fluorapatites ("staffelite" from Staffel, Germany, and "francolite" from Cornwall, England).

Table 1. Comparison of *X*-ray Data

	Plane indices	2θ (mm.)	*d*-spacings (Å)	Δ[1] (mm.)
Hydroxylapatite	231	50.6	1.804	2.7
	004	53.3	1.719	
Fluorapatite	231	50.8	1.797	2.5
	004	53.3	1.719	
Carbonate-fluorapatite	231	50.9	1.794	2.3
	004	53.2	1.722	

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[1] Difference between 2θ-values (in mm.) for lines (231) and (004).

The presence of calcite in artificial calcite-apatite mixtures was readily discernible. Powder films were taken of mixtures of a pure fluorapatite from Durango and chemically pure calcium carbonate, with the latter present in proportions of 2, 3, 4, 5, 7, and 10 per cent by weight. The x-ray pattern of the mixture containing 2 per cent calcium carbonate was indistinguishable from that of pure fluorapatite. Three characteristic calcite lines (d-values of 3.038 Å, 2.089 Å, and 1.914 Å) were identified in each of the remaining mixtures. The presence of calcite in amounts of 3 per cent and above is thus detectable in artificial mixtures by use of the x-ray method described.

Differential Thermal Analysis

It is unlikely that carbonate would be held by identical bonding forces in both calcite and carbonate-apatite. Consequently the heat and temperature of decomposition of calcite should differ from those of carbonate-apatite. These properties are conveniently measured by means of a differential thermal analysis apparatus. The theoretical and operational details of this apparatus have been described by Hendricks, Goldich, and Nelson (1946, pp. 64–76).

The following investigations were performed on a portable field unit[1] that was rewired for use with platinum-platinum (90%), rhodium (10%) thermocouples.

Reference curves were prepared from the thermal data for chemically pure calcium carbonate, calcite, fluorapatite, carbonate-fluorapatite, and artificial mixtures of a pure natural fluorapatite and 2, 5, 7, and 10 per cent chemically pure calcium carbonate. The sharp and abrupt endothermic peaks resulting from the thermal decomposition of both natural calcite and chemically pure calcium carbonate are comparable to those reported in the literature (Cuthbert and Rowland, 1947, p. 113; Faust, 1950, p. 217). Fluorapatite alone gives a smooth curve when heated to 950° C. The carbonate-fluorapatite, an optically homogeneous mineral from Staffel, Germany, containing 3.1 per cent CO_2, which is equivalent to 7 per cent $CaCO_3$, did not show the distinct endothermic peak typical of calcite. Instead it gave a smooth curve, which, in relation to the fluorapatite curve, suggests a very gentle endothermic trend over the entire temperature range. These observations are in agreement with the heat-loss data reported by Geiger (1950, pp. 168, 178).

Each of the artificial mixtures of calcium carbonate and fluorapatite gave a calcite peak superimposed on the smooth fluorapatite curve. The relative heights and breadths of peaks for the different mixtures are presented in Table 2. The relative areas included under the calcite peaks, which were measured with a polar planimeter, are roughly proportional to the amounts of calcium carbonate present. The corresponding variations of peak temperatures with amount of calcium carbonate present are due primarily to differences in the partial pressures of the CO_2 liberated during the early stages of thermal decomposition (Rowland and Lewis, 1951, pp. 86–87).

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[1] Designed by R. A. Nelson, of the U. S. Geological Survey, and manufactured by the Eberbach & Son Co., Ann Arbor, Mich.

In order to check the validity of the results, G. T. Faust was kind enough to run pure fluorapatite, carbonate-fluorapatite, and the mixture of fluorapatite and 2 per cent calcium carbonate on a more sensitive differential thermal unit (Faust, 1948, pp. 337–345). The curves obtained with our portable field unit compared favorably with those from the more precise instrument, and we ascertained that the portable unit could be depended upon to detect a minimum of 1.6 per cent calcite in natural phosphates.

TABLE 2. DIFFERENTIAL THERMAL DATA FOR ARTIFICIAL MIXTURES OF CALCIUM CARBONATE AND FLUORAPATITE

Material	Per cent CO_2 present as calcite	Calcite peak data			
		Peak temperature (°C.)	Relative peak height	Peak breadth[1] (°C.)	Area under peak (relative)
Fluorapatite[2]	0.0	No calcite peak			
Fluorapatite[2]+2% $CaCO_3$	0.88	780°	1.3	55°	18
Fluorapatite[2]+5% $CaCO_3$	2.20	810°	2.4	50°	29
Fluorapatite[2]+7% $CaCO_3$	3.08	830°	3.9	55°	58
Fluorapatite[2]+10% $CaCO_3$	4.40	835°	4.7	110°	79
Chemically pure calcium carbonate	44.00	880°	16.6	110°	417

[1] Measured at the base of the peak in degrees centigrade.

[2] Fluorapatite from Ontario, Canada.

DIFFERENTIAL SOLUBILITY TESTS

Differential solubility methods have been applied successfully in the past to the separation of the components of a mixture. The carbonate-apatite problem is principally one of distinguishing a homogeneous from a heterogeneous mineral composition. The applicability of differential solubility tests to this problem depends on the existence of a solvent in which carbonate-apatite is relatively insoluble as compared with calcite.

A solvent that fulfills this requirement is a half-molar aqueous solution of triammonium citrate. The solubility of calcite in this solvent has been established by Rindell (1910, pp. 452–458).

The data in Table 3 indicate that calcium carbonate is approximately 50 times as soluble as either fluorapatite or carbonate-fluorapatite in this reagent.

TABLE 3. SOLUBILITIES IN 0.5 M TRIAMMONIUM CITRATE SOLUTION

Material	Solubility in grams/100 ml. solvent
Calcium carbonate, cp	0.660 g. ±0.060
Fluorapatite, Durango, Mexico	0.014 g. ±0.000
Carbonate-fluorapatite, Staffel, Germany	0.012 g. ±0.003

The triammonium citrate solution was prepared by titration of chemically pure diammonium citrate with ammonium hydroxide to pH of 8.1 ± 0.1. The solubility values in Table 3 were determined by the addition of 100 ml. of solvent to 1 g. of powdered sample (ground to pass 200 mesh) in a glass-stoppered Erlenmeyer flask. The mixture was permitted to stand for 24 hours at room temperature, with intermittent shaking, after which it was filtered and the filtrate ignited to constant weight. The solubility of pure calcium carbonate was computed from the weight of the ignited product (calcium oxide).

Geiger's heat-loss data (1950, p. 168) indicate that "staffelite" loses at least 65 per cent of its CO_2 content by ignition to 900° C. The carbonate-fluorapatite solubility value given in Table 3, however, required no correction for CO_2 lost during ignition as the maximum possible loss is less than 0.0004 g., which is beyond the limiting precision of the measurements.

On the basis of solubility values obtained by 48- and 72-hour treatments, which were identical with the solubility values obtained in 24 hours (see Table 6), it was concluded that any discrepancies due to incomplete solution were not large enough to invalidate the conclusions reached by these measurements. In Rindell's original work (1910, pp. 452–458), it was assumed that equilibrium is attained in 24 hours.

The relatively high limits of error reported in Table 3 were undoubtedly due to the lack of precisely controlled conditions. The precision of these measurements can be improved by use of a controlled-temperature bath, of a constant stirring mechanism, and by very precise standardization of the solvent. The degree of precision attained, however, was sufficient for the immediate purposes of these investigations.

The calcite content of each phosphorite was determined by treating each 1 g. sample with 100 ml. of solvent in the manner described for the solubility measurements. After the mixture was filtered the insoluble residue was washed with distilled water. The dried insoluble residue and the original sample were analyzed gravimetrically for carbonate content by absorption of carbon dioxide in ascarite (Kolthoff and Sandell, 1948, pp. 385–389) after liberation of the carbon dioxide by dilute hydrochloric acid. The difference between the carbon dioxide content of the original sample and that of the leached residue was taken as a measure of carbon dioxide present as calcite. The results of the absorption method are reproducible to 1 mg.; the carbon dioxide content for 1 g. of sample is therefore reliable to 0.1 per cent

In order to establish the effectiveness of the solvent on calcite in the presence of apatite, artificial mixtures of the Durango fluorapatite containing 2, 5, 7, and 10 per cent of chemically pure calcium carbonate were treated with the solvent. In each of these check determinations the insoluble residues were found to be entirely free of calcite within the limits of error.

ANALYSES OF NATURAL PHOSPHORITES

These methods, whose reliabilities have been established for artificial mixtures and pure end members, were then applied to natural phosphorites.

Samples were selected from the phosphatic shale member of the Phosphoria formation (Idaho and Utah) on the basis of their carbonate content, as shown in Table 4.

In addition, a fossil rib bone (genus *Felsinotherium*) of probable early Pliocene age (sample 74-*M*-4) from the Boyette mine, Hillsborough County, Fla., was included in these analyses.

TABLE 4. CARBON DIOXIDE CONTENT OF PHOSPHORITES

With visible calcite		Without visible calcite	
Sample no.	CO_2 content (per cent)	Sample no.	CO_2 content (per cent)
150 (Conda mine, Idaho)	4.7	311*C* (Trail Canyon, Idaho)	1.9
163 (Conda mine, Idaho)	4.5	309 (Trail Canyon, Idaho)	1.6
161 (Conda mine, Idaho)	3.1	317 (Trail Canyon, Idaho)	1.3
164*B* (Conda mine, Idaho)	2.5	300 (Trail Canyon, Idaho)	1.2
1007-1 (Laketown, Utah)	1.8	315*B* (Trail Canyon, Idaho)	1.1
164*A* (Conda mine, Idaho)	1.4		
1007-7 (Laketown, Utah)	1.4		

A compilation of the results obtained by the three methods appears in Table 5. The samples are arranged in the order of increasing calcite content as determined by the solubility method (column 3).

The differential thermal data in columns 4 and 5 show an orderly progression of increasing temperatures and endothermic peak heights with increasing calcite content for the samples containing more than 0.7 per cent CO_2 combined as calcite.

In addition to calcite, the phosphorites contain small but varying amounts of thermally active substances, chiefly clays, organic matter, and iron oxides. Of these materials, certain clays may undergo thermal changes in the temperature region of the calcite decomposition, thereby interfering with the endothermic peak due to calcite alone. For most of the materials examined, this interference was not great enough to cause a serious discrepancy in the calcite peak measurements. Of the group of samples in Table 5, sample 163 has the lowest phosphate content; accordingly the relative proportions of foreign materials is greater; and the contaminating substances have interfered by causing a broadening of the calcite peak at its base, which has resulted in an abnormally large area under the peak. Where the above-mentioned thermally active sub-

TABLE 5. COMPARISON OF RESULTS OBTAINED BY SOLUBILITY, DIFFERENTIAL THERMAL, AND *X*-RAY METHODS ON NATURAL PHOSPHATE MATERIALS

Sample	Total CO_2 content (per cent) in unleached sample	Per cent CO_2 present as calcite[1]	Differential thermal analysis Calcite peak data				*X*-ray data	
			Peak temperature (° C.)	Relative peak height	Peak breadth[2] (° C.)	Area under peak (relative)	Calcite lines	Δ[3] (mm.)
Carbonate-fluorapatite[4]	3.1	0.0	No calcite peak				Absent	2.3
315*B* (Trial Canyon, Idaho)	1.1	0.0	No calcite peak				Absent	2.3
74-*M*-4 (Boyette mine, Fla.)	2.9	0.0	No calcite peak				Absent	2.3
311*C* (Trial Canyon, Idaho)	1.9	0.1	No calcite peak				Absent	2.5
300 (Trial Canyon, Idaho)	1.2	0.2	No calcite peak				Absent	2.4
318 (Trial Canyon, Idaho)	1.3	0.2	No calcite peak				Absent	2.3
309 (Trial Canyon, Idaho)	1.6	0.2	No calcite peak				Absent	2.3
317 (Trail Canyon, Idaho)	1.3	0.2	No calcite peak				Absent	2.4
164*A* (Conda mine, Idaho)	1.4	0.4	No calcite peak				Absent	2.3
1007-7 (Laketown, Utah)	1.4	0.4	No calcite peak				Absent	2.3
1007-1 (Laketown, Utah)	1.8	0.6	No calcite peak				Absent	2.3
162 (Conda mine, Idaho)	2.3	0.7	775°	0.5	22°	5	Absent	2.3
164*B* (Conda mine, Idaho)	2.5	1.0	785°	1.2	30°	18	Absent	2.3
161 (Conda mine, Idaho)	3.1	1.4	790°	1.9	60°	34	Absent	2.3
163 (Conda mine, Idaho)	4.5	3.1	800°	4.3	95°	99	Present	2.2
150 (Conda mine, Idaho)	4.7	4.0	835°	6.7	60°	70	Present	2.3

[1] Results of triammonium citrate solubility tests.

[2] Measured at the base of the peak in degrees centigrade.

[3] Difference between 2θ-values (in mm.) for lines (231) and (004).

[4] From Staffel, Germany.

stances are comparatively negligible, as in sample 150, where they are essentially absent, the resulting thermal curves are almost identical with those obtained from the artificial calcite-apatite mixtures.

The presence or absence of the three calcite lines in *x*-ray powder films (Table 5, column 8) depends to a certain extent on the quality of the pattern. The artificial mixture of fluorapatite and 3 per cent calcium carbonate gave a sharp pattern in which the calcite lines could be detected. Sample 161, a phosphorite that contains 3.2 ± 0.2 per cent calcite as determined by the solubility tests, gave a less distinct pattern in which the calcite lines were not visible. The *x*-ray pattern obtained from sample 163, a phosphorite containing 7.0 ± 0.2 per cent calcite, showed the same poor definition and high background intensity as that obtained from sample 161, but in this case the calcite lines were detectable.

The last column lists the differences between the 2θ-values for lines (231) and (004) referred to in Table 1.

Samples from the Conda mine in southeast Idaho are unweathered, black, and dense, whereas those from the nearby outcrop at Trail Canyon are weathered, brown, and less consolidated. Both groups of samples are from equivalent parts of the Phosphoria formation. This correlation is based on the distinctive character of a single bed of brecciated nodules of phosphatized palaeoniscoid fish scales and small coprolitic masses (?) identified by D. H. Dunkle, of the U. S. National Museum. At the Conda mine (sample 150) this bed contains 4.1 per cent of free calcite, whereas at Trail Canyon (sample 300) the calcite content is negligible. The characteristically low calcite content of the Trail Canyon samples as compared to that from the Conda mine is probably due to natural leaching processes. The other samples from Trail Canyon and the Conda mine are "pelletal phosphorites" typical of the phosphatic shale member of the Phosphoria formation in southeast Idaho.

All samples contain at least 75 per cent apatite. The average organic content of the Conda mine samples is higher than that of the Trail Canyon samples by about 3 per cent. The samples from the Laketown district, Utah, are sieve fractions of one bed that contains visible calcite, detrital material, and secondary clays but is devoid of organic matter.

SPECIAL CONSIDERATIONS

Hendricks and Hill (1950, p. 737) have concluded from investigations of the mineral constituents of bone that "carbonate is present on entrapped surfaces and not as constituent within the lattice of the mineral francolite, the typical 'carbonate apatite' . . . ." If the carbonate present in these samples is present as calcite entrapped at crystallite boundaries it is possible that some part of the included calcite would not be accessible to the solvent. The following experiments were designed to test the availability of calcite occluded along a continuous, as well as along a discontinuous, network of surfaces.

Two samples, 150 (Conda) and 162 (Conda), containing 4.7 per cent and 2.3 per cent CO_2, respectively, were treated with the solvent, a half-molar solution of triammonium citrate, for 24, 48, and 72 hours. The carbon dioxide content of the leached residues was essentially constant, as shown in Table 6.

TABLE 6. CARBON DIOXIDE CONTENT OF RESIDUES OF SAMPLES 150 AND 162 AFTER LEACHING FOR VARIOUS LENGTHS OF TIME

Sample	Carbon dioxide content (per cent) after leaching for:		
	24 hours	48 hours	72 hours
150 (Conda mine)	0.9±0.0	0.9±0.0	0.9±0.0
162 (Conda mine)	1.6±0.1	1.6±0.0	1.5±0.1

These results indicate that equilibrium between the solid phases, consisting mainly of apatite, and the liquid phase, containing dissolved calcium carbonate, dissolved apatite, and solvent is established after 24 hours. Preliminary solubility measurements (Table 3) show that 100 ml. of solvent, saturated with apatite, can dissolve more than 0.6 g. of calcium carbonate. One-gram samples of 150 and 162 contain sufficient carbon dioxide to account for 0.11 g. and 0.05 g. of calcium carbonate, respectively. The liquid phase in a mixture of 1 g. of either of these materials and 100 ml. of solvent, in equilibrium with the solid phase, is unsaturated with respect to $CaCO_3$. Under these conditions no solid calcium carbonate should exist in contact with the liquid.

If the carbonate is restricted to a continuous network of calcite along entrapped surfaces, the calcite would be accessible to the solvent only at the points where the network was exposed at the surface of the particle. Under these conditions the amount of calcite dissolved by the solvent should vary with the length of time. The absence of such variation (Table 6) is evidence against a continuous network of occluded calcite.

Additional evidence comes from a study of the relation of the solubility of carbonate-apatite to particle size. The carbonate-fluorapatite, an optically homogeneous mineral from Staffel, Germany, contains 3.1 per cent CO_2, which is equivalent to about 7 per cent calcium carbonate. The solubility measurements (Table 5) indicate that no calcium carbonate is present. The carbonate content is therefore present as a constituent of the apatite structure, or as some other form of carbonate that is not combined as calcite. Three sieve fractions (+60 −84, +165 −200, and +325 −400) of this mineral were treated with the solvent for 24 hours. The relative surface areas per given weight of material have been computed for each sieve size. These computations, which take into account the columnar shape of the fragments (length of columns are approximately twice their width), give the values listed in Table 7.

TABLE 7. RELATIVE SURFACE AREAS FOR DIFFERENT PARTICLE SIZES OF CARBONATE-FLUORAPATITE

Sieve size	Relative surface area
+ 60 − 84	1.00
+165 −200	2.86
+325 −400	6.44

In each size fraction no difference in the CO_2 content of the leached residue as compared with that of the unleached material was detectable within the limits of measurement.

The volume of a single particle of the smallest size group (+325 −400 mesh) is 1.3×10^{-7} cm.^{3}, and it represents more than a sixfold increase in surface area per unit weight of material over that of the coarsest group (+60 −84 mesh). Regardless of the dimensions of "entrapped" calcite, if the 3.1 per cent CO_2 content of this carbonate-fluorapatite is present as calcite, the calcite must occupy 9.2 per cent of the total volume. To the extent of the increase in surface area represented by the difference between the coarsest and finest particle sizes considered here, the experimental observations constitute an argument against occluded calcite along a discontinuous network. Furthermore, if any carbonate is present as occluded calcite, its distribution in this mineral must be random. The solubility relations of this mineral to half-molar triammonium citrate solution are generally those expected from a single, very slightly soluble, homogeneous compound.

CONCLUSIONS

The presence of calcite in carbonate-bearing phosphorites has been determined by use of *x*-ray methods, differential thermal analyses, and solubility tests. The minimal detectable amounts of calcite, as shown by the results in Table 5, are 7.0 per cent, 1.6 per cent, and 0.1 per cent, respectively, for the above methods.

Application of these procedures leads to the conclusion that in some samples (carbonate-fluorapatite from Staffel, Germany; 74-M-4; and

315B, Trail Canyon) no part of the CO_2 content, which ranges from 3.1 to 1.1 per cent, is present as calcite. The other phosphorites examined in this survey contain variable amounts of calcite, but in all of them an appreciable part of the carbonate is not present in the form of calcite.

The results of solubility tests, in which the particle size of sample and the length of solution time were varied, imply that the carbonate content is not due to shielded calcite entrapped along continuous or discontinuous surfaces.

APPENDIX

EXPOSURE OF CALCITE OBSCURED BY ORGANIC MATTER IN PHOSPHATE ROCK

JEANNE D. WEISER

Small amounts of calcite in some samples of phosphate rock from southeastern Idaho are often concealed in thin sections or powder mounts by the high content of organic matter, and no clue to the presence of the calcite is offered by chemical analysis for total CO_2 because the major mineral constituent is a carbonate-bearing apatite. By the slight modification and combination of two well-known petrographic techniques, the presence and distribution of calcite and the reason for its obscurity were made clear.

Rectangular blocks $1\frac{1}{2}$ inches by 1 inch by 1 inch of black phosphatic rock were cut and placed in small porcelain crucibles so that the blocks were resting only on corners and edges. They were then placed in a Tempco Electric Furnace and heated through three temperature stages as follows: 1 hour with the temperature control set at "low" (from room temperature to 200° C.), $1\frac{1}{2}$ hours at "medium" (from 200° C. to 425° C.), and $1\frac{1}{2}$ hours at "high" (from 425° C. to 775° C.). At the end of this time the blocks turned white.

Slow and gradual heating prevented the samples from decrepitating because of sudden loss of water, and keeping the temperature below 800° C. avoided any appreciable destruction of the calcite. It is possible that calcite near the outer surfaces of the blocks calcines at this temperature, as the dissociation meets progressively less interference nearer the surface.

Uncovered thin sections were prepared from the blocks roasted in this manner and then were stained by the silver nitrate-potassium chromate method described by Krumbein and Pettijohn (1938, p. 496).

The thin sections turned a grayish orange pink (5YR 7/2), a color that can be resolved by means of the petrographic microscope into microscopic spots of reddish brown calcite fairly evenly distributed throughout the cryptocrystalline phosphate pellets.

When these factors concerning the calcite are considered, that is, its relative paucity compared with the organic matter, its small size, and even distribution, it is understandable that more than 2 per cent of this highly birefringent mineral could be rendered invisible.

The precise quantitative method for the detection of calcite in the presence of carbonate-bearing apatite, described in the main body of this report, was subsequently developed so that the chief value of this experiment now lies in exposing the distribution of calcite hitherto hidden by organic matter in phosphate rocks. It is also reassuring to observe that the silver nitrate-potassium chromate method of staining will operate on particles of very small size.

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COORDINATION MODELS

Cecil J. Schneer, *Hamilton College, Clinton, N. Y.*

Abstract

Models of the atomic structures of minerals have been made by considering the structures as assemblages of coordination polyhedra. Tetrahedra and octahedra are the most important coordination polyhedra for mineral structures. Close packed space may be considered as composed of octahedra and tetrahedra, the corners of which may represent the centers of close packed spheres. The polyhedra are in the ratio of two tetrahedra to one octahedron for every close packed sphere. For close packed ionic structures, the centers of mass of anions lie at the corners of the polyhedra, and centers of polyhedra may be occupied by cations or vacant, the proportions of occupied to vacant polyhedra being determined by the chemical formula. In the models, vacant polyhedra are omitted. The method may be extended to non-close packed structures. One size of tetrahedral block and two sizes of octahedral blocks are sufficient to build models of the main framework of nearly all mineral structures. Angles of linkage of polyhedra are regular and evident on inspection.

Introduction

Models of the type described in this paper have been constructed by Pauling[1] and others. However, as far as the author knows, there is no published account of their construction or indeed any specific account of coordination theory as related to models of the atomic structures of minerals. The models here designated coordination models were constructed in the elementary class in mineralogy at Hamilton College. Because of the simplicity of these models and the ease with which they may be assembled, it is possible to use their construction for a systematic introduction to the atomic structures of minerals. Briefly, structures are conceived as assemblages of regular polyhedra, the corners of which represent the centers of mass of anions, and the centers of which represent coordinated cations. Blocks of appropriate size and shape are used as units of construction. The models represent graphically Pauling's principles[2] governing the structures of ionic crystals. They are concerned with the geometrical and electrical relationships between units of structure. Standard methods of constructing models of the atomic structures of minerals such as the packing models developed at Massachusetts Institute of Technology[3] are, because of the expense and complexity of construction involved, beyond the reach of the elementary student. Moreover, the coordination models specifically point the student towards coordination theory. They reduce the complex geometry of atomic structures to relatively simple patterns involving simple forms.

[1] Pauling, L., The nature of the chemical bond, Ithaca (**1939**), Figs. 48-2, 6, 9.

[2] Pauling, L., *J.A.C.S.*, **51,** 1010 (1929).

[3] Buerger, M. J., and Butler, R. D., *Am. Mineral.*, **21,** 150–172 (1936).

Modern coordination theory has its origin in the brilliant postulate of the tetrahedral orientation of the four bonds of carbon by van't Hoff and le Bel in the last century. In this century, W. L. Bragg determined structures based on the close packing of oxygen. The fundamental units of ionic structures are visualized as approximately spherical and semi-rigid with characteristic radii and electrical properties. Spheres when packed together so as to occupy the least space (close packed) will enclose amongst themselves, two kinds of openings. If we consider lines joining the centers of close packed spheres, it is evident that within any layer, lines joining the centers of three adjacent spheres enclose an equilateral triangle. If a single sphere of a second layer is placed in contact with the first three adjacent spheres, the lines joining the centers of all four spheres will enclose four equilateral triangles making a regular tetrahedron. Any sphere of radius appropriately smaller than the radius of the close packed spheres might fit in the interstice between the four close packed spheres and would thus be tetrahedrally coordinated. When the array of close packed spheres is extended indefinitely in three dimensions, the ratio of the number of tetrahedral interstices to the number of close packed spheres will be two to one. Still another type of opening exists surrounded by six close packed spheres. The triangles formed by the center to center lines of the six spheres bound a regular octahedron. In extended array, the number of octahedral openings is exactly equal to the number of close packed spheres. Thus, considering the centers of spheres only, close packed space consists of tetrahedra and octahedra in the ratio of two to one. This is a geometrical property of close packed space directly analogous to the hexagonal jointing in columnar basalts.

In inorganic structures, oxygen and less commonly other ions may be found in close packed array. Assuming close packing of oxygen, consideration of the chemical formula for forsterite, Mg_2SiO_4, shows that there is not a sufficient number of cations to fill the available interstices. Therefore the structure will consist of occupied and unoccupied polyhedra. Crystals are built up by a regular repetition of identical groups of atoms or ions and therefore the alternation of these occupied and vacant polyhedra must be systematic and orderly. If Mg atoms are coordinated octahedrally and Si atoms tetrahedrally, only one out of every two octahedral openings and one out of every eight tetrahedral openings will be occupied. Not many arrangements will be found to fit these fundamental requirements. Space group theory imposes no limitations on the number of structures beyond those of symmetry. In a discussion of the 230 space groups, Phillips states, "the *actual* number of different arrangements possible is, of course infinite . . . ."[4] Coordination theory is an attempt to reduce this degree of empiricism. Thermodynamic considerations lead Pauling[5] to assume that the octahedral and tetrahedral groupings are a function of the ionic radii; that they persist even when the anions are not in closest packing; and that each ion must be electrically satisfied within a reasonably short distance. Cast in the form of principles governing the structures of ionic crystals, these assumptions are the basis of modern coordination theory.

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[4] Phillips, F. C., An introduction to crystallography, London (1946), p. 235.

" . . . we find that very few alternative structures which obey Pauling's law remain open to a mineral of given composition, and one of these alternatives always turns out to be the actual structure of the mineral."[6]

DIADOCHY

Diadochy, or the ability of ions or atoms to substitute for other ions or atoms in a given structural position[7] is subordinated in the coordination models. The models show structural positions and emphasize the structural limitations on diadochy.

GENERAL PROCEDURE

The models so far constructed at Hamilton College are of paper, cut and folded to form individual blocks which are then assembled.[8] Tetrahedra and octahedra, 4 cm. on a side, and octahedra 4.6 cm. on a side are used. When these represent polyhedra of oxygen surrounding cations such as Al''', Si'''', Mg'', and Fe'', the scale is approximately 1.5 cm. equals 1 Angstrom, or a magnification of 1.5×10^8. A side on the larger octahedra will just bridge the apices of two tetrahedra whose bases join at 120° angles in a common plane. Individual blocks may be painted with colors indicating the nature of the coordinated cation. The blocks are assembled permanently with collodion or any other quick drying cement.[9]

A description of the required structure may be obtained from a number of sources.[10,11] Ordinarily the coordination of the cations will be stated as well as illustrated. Silicon, aluminum, beryllium, boron, zinc, iron, magnesium, and manganese may be tetrahedrally coordinated by oxygen. Aluminum, iron, magnesium, manganese, cobalt, nickel, chromium, titanium, lithium, molybdenum, tungsten, calcium, zinc, and cadmium may be octahedrally coordinated.[12] Large ions such as the alkali metals coordinated by more than six anions are either omitted from the models or are represented by balls mounted on inflexible rods. Small ions coordinated by less than four anions are likewise omitted or are represented by balls. The tetrahedra and octahedra are arranged so as to satisfy the requirements of Pauling's principles. Adjacent polyhedra may share corners, edges, or faces. If polyhedra share a corner, then the charge on the anion represented by the corner is shared equally by the centrally coordinated cations of the polyhedra. If polyhedra share an edge, then two corner anionic positions are common to the centrally coordinated cations. If two polyhedra share a face, then three anionic positions are common to the two centrally coordinated cations. The number of polyhedra common to a single corner anionic position represents the coordination of the anion by cations. This linkage should be clear in the description of the structure. It may be helpful to make a tracing from a diagram showing the positions of atoms, and on the tracing, connect the anions about each cation by straight lines. This should yield a block diagram of the desired model. When the positions of the blocks have been determined, they are glued together. The angular relationships between blocks are in every case simple and evident on inspection. As will be apparent in the description of the models below, use of regular octahedral and tetrahedral blocks leads to idealization of the structures.

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[5] Pauling, L., *op. cit.*

[6] Bragg, W. L., Atomic structure of minerals, Ithaca (**1937**), p. 36.

[7] Strunz, H., *Zeits. Krist.*, **96**, 7 (1937).

[8] Blocks in hard, colored plastic are being developed.

[9] Several cements with acetone and ether as solvent were tried and found satisfactory. Ordinary mucilage was also satisfactory. The contiguous parts are moistened with cement and held in position while the cement dries. Pins protruding from the vertices were tried and found unnecessary.

[10] Bragg, W. L., *op. cit.*

[11] Wyckoff, Ralph W. G., The structure of crystals, New York (**1931**).

When the main principles of the structure are clear, groups of units may be assembled separately and then fitted together. For example, chains of polyhedra are used to construct models of the pyroxenes; double chains of tetrahedra are used to construct models of the amphiboles.

POLYMORPHS OF TiO_2

Figure 1*a* shows a model of anatase, TiO_2, $I4/amd$[13], illustrating the octahedral coordination of Ti'''' with three octahedra meeting at every corner (O). The structure has been idealized by taking as equal, all distances between adjacent oxygens. This is a simplification but not a serious one. In actuality, the oxygen to oxygen distance is reduced slightly if the same pair of oxygens is common to more than one octahedron (an edge is shared), while the distance is increased by the mutual repulsion of the oxygens where the edge is not shared.

Two octahedra are glued together at a corner. A third octahedron is glued between the first two sharing an edge with each. This last octahedron is the central block of the model. A second pair of octahedra sharing a corner is glued beneath the central octahedron in the same manner as the first pair but turned 90 degrees away from the line of the first pair. Since in this structure, all Ti'''' ions occupy equivalent positions, any block may be treated as the central block, and pairs of octahedra disposed above and below it to extend the model. The model in Fig. 1*a* was completed by surmounting the upper and lower pairs of octahedra by square rings of four octahedra. These rings amount to two additional pairs of octahedra each sharing edges with one of the octahedra of the first pairs.

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[12] Bragg, W. L., *op. cit.*, p. 33.

[13] Vegard, L., *Phil. Mag.*, **32**, 65 (1916).

Figure 1*b* is the model of brookite, TiO_2, P*bca*.[14] Again each Ti'''' ion is octahedrally coordinated by oxygen and each oxygen is common to three Ti'''' ions.

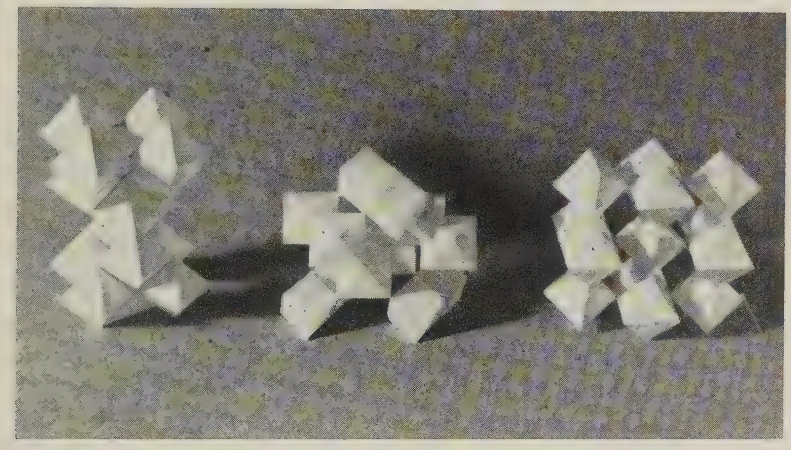

FIG. 1. Polymorphs of TiO_2. (*a*) The unit cell of anatase. The *c* axis is vertical. (*b*) The structure of brookite viewed along the *c* axis with the *a* axis vertical. The unit cell would be complete without the left pair of octahedra on each of the first and third layers from the bottom. (*c*) The structure of rutile viewed along the *c* axis. Lines joining the centers of the paired octahedra enclose the unit cell.

This model also represents an idealized structure. A pair of octahedra are glued together along an edge. Six such pairs are required. Two pairs are aligned on a flat base so that forward faces slope down directly towards the observer. These are the first layer in Fig. 1*b*. A third pair of octahedra is inverted so that forward faces slope up towards the observer. This is the second layer. It bridges the two pairs of the bottom layer. Two pairs make up the third layer. They are oriented like the pair in the second layer with forward faces sloping up towards the observer,

[14] Pauling, L., and Sturdivant, J. H., *Zeitz. Krist.*, **68**, 239 (1928).

but the alignment of each third layer pair is 60° away from the alignment of the second and first layer pairs. If the model is extended along the *c* axis, all the pairs of octahedra will become parallel zigzag chains.

The fourth layer is begun as a single pair bridging the pairs of the third layer. The octahedra in the fourth layer are oriented like those of the first layer but the pair is aligned parallel to the third layer pairs.

Figure 1*c* is the well known rutile structure, TiO_2, $P4/mnm$[15] viewed along the four fold axis and idealized. Four pairs of octahedra joined together on edges are required. These are joined to a single octahedron in the center as shown in Fig. 1*c*. Four octahedra are then added, one in each corner, each oriented parallel to the central octahedron and each linking two of the four pairs. Note that the corners of each pair of octahedra are connected by the edge of a single octahedron. Note also that the octahedra of the figure may be repeated indefinitely along the line of sight and that each corner octahedron may be considered as a central octahedron in an extended model.

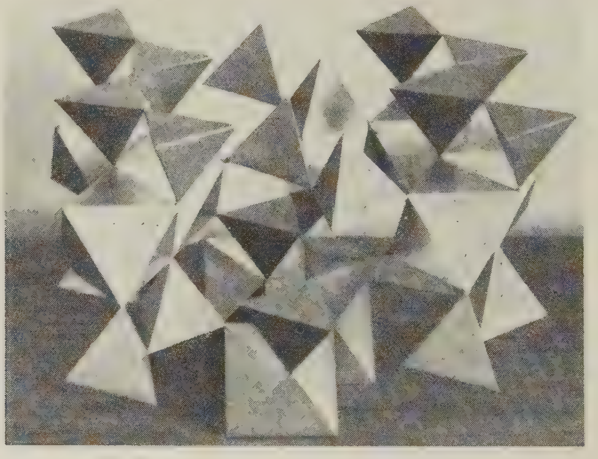

FIG. 2. The structure of diopside. The chains of tetrahedra are parallel to the *c* axis. Note the vertical *c* glide plane perpendicular to the page. The unit cell is only two tetrahedra long in the *c* direction and two thirds of the length illustrated along the *b* axis (from left to right). An additional layer (two chains) of tetrahedra along the *a* axis (linked to the bottom corners of the central octahedral chain) would complete the unit cell in that direction.

DIOPSIDE

Figure 2 is a reproduction of a photograph of a model of a portion of the unit cell of diopside, $CaMgSi_2O_6$, $C2/c$,[16] showing the linkage between chains of silica tetrahedra and magnesia octahedra. Calcium atoms (not shown) occupy the irregular octahedral embayments between the magnesia chains and the apices of the silica tetrahedra.

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[15] Vegard, L., *op. cit.*

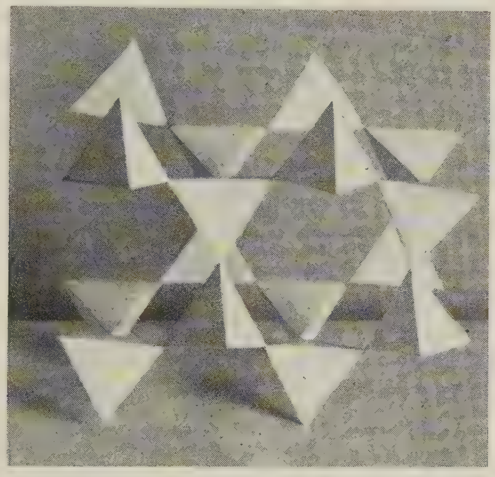

FIG. 3. The structure of tridymite viewed along the *c* axis. The eight tetrahedra in front were assembled as a unit. The eight tetrahedra behind are a reflection of the first unit, joined to it along apices. The unit cell would be complete without the four tetrahedra on the left side of the model.

TRIDYMITE

Figure 3 is a model of tridymite, SiO_2, $C\bar{6}2c$ or $C6/mmc$,[17] built by Joseph Hull. This illustrates the extended linkage of the SiO_2 minerals. The tetrahedra are assembled point to point in hexagonal rings with apices pointing alternately up and down. One ring is fitted on top of another, apex to apex. By extension in three dimensions, the network structure is developed. There are two ways of fitting the rings together apex to apex, the one illustrated by the model and an alternate. The alternate assembly is the structure of cristobalite, SiO_2, $Fd3m$(?). Higher symmetry should be characteristic of higher temperature, and cristobalite is the high temperature form of SiO_2 next above tridymite.

Our experience at Hamilton College has been that these models greatly simplify the study of mineral structures. We have found the assembly of these models to be well within the abilities of beginning students in mineralogy.

Manuscript received July 21, 1951

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CRYOLITE TWINNING

J. D. H. DONNAY, *The Johns Hopkins University, Baltimore 18, Maryland.*

ABSTRACT

Thirteen twin laws are predicted by theory (Friedel); most of them are observed (Böggild). The twin operations are the operations of pseudo-symmetry of a quasi-cubic double cell, obtained from the cryolite cell by transformation $1\bar{1}0/110/001$.

INTRODUCTION

This note is intended as a preamble to Dr. Wrinch's paper, which follows it. It summarizes the status of the cryolite problem, which I studied a number of years ago at the request of Professor Charles Palache.

Friedel's treatment of twinning (1905, 1926), applied to cryolite, leads to the prediction of 13 twin laws. We may use Krenner's axial elements for cryolite,

$$a:b:c = 0.966:1:1.388, \qquad \beta = 90°11\,,$$

which have been confirmed by x-rays

$$5.46:5.61:7.80 = 0.973:1:1.390, \qquad \beta = 90°11\,, \qquad P2_1/n,$$

(Náray-Szabó and Sasvári, 1938). The condition that must be satisfied for twinning to occur is that the ratios $a^2:b^2:c^2:\text{ca}\cos\beta$ approach rational numbers. It is here obeyed, as

$$a^2:b^2:c^2:\text{ca}\cos\beta = 0.934:1:1.927:-0.004 \cong 1:1:2:0.$$

Twins are to be expected.

The twinning is controlled by the pseudo-symmetry of a double cell, which can be defined (Fig. 1) by the vectors

$$\boldsymbol{A} = \boldsymbol{a} - \boldsymbol{b}, \qquad \boldsymbol{B} = \boldsymbol{a} + \boldsymbol{b}, \qquad \boldsymbol{C} = \boldsymbol{c}.$$

This cell is a pseudo-cube, for $A = B$ is very nearly equal to C. Any operation of cubic pseudo-symmetry of this double cell can be a twin opera-

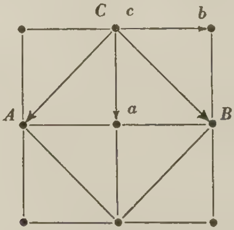

FIG. 1. Net plane (001) of the cryolite lattice showing four meshes (***ab***) and one double mesh (***AB***).

tion, unless it happens to be an operation of the monoclinic symmetry of the crystal. This is a case of "twinning by reticular pseudo-merohedry" (Friedel, 1905, 1926), that is to say, one in which a multiple lattice simulates a symmetry higher than that of the lattice. In cryolite the double cell ***ABC*** is *C*-centered. Leaving out the *C*-centering node, this cell becomes primitive and can be used to define a pseudo-cubic lattice, which is called a "double lattice" of cryolite because its cell has twice the volume of the cell in the true cryolite lattice.

To predict all the possible twin laws, I shall proceed as follows: Consider each element of cubic pseudo-symmetry of the cell ***ABC***, designating it by its symbol referred to Krenner's axial elements. Find out what effect the monoclinic symmetry may have on its becoming a twin element. Finally, for each predicted twin law, I shall refer by number to a list of the observations on record (see below).

Theoretical Predictions

The vertical 4-fold axis of the pseudo-cubic cell is [001]. It is not an element of the monoclinic symmetry, and it may therefore become a twin axis. It can give rise to two distinct twin laws according as the angle of rotation is 90° (see observation No. 11) or 180° (see observation No. 6). Note that the 270° rotation does not yield a distinct twin law, for a twin in which individual I is brought to coincidence with individual II by a clockwise rotation through 270° would be indistinguishable from a twin in which individual II can be made to coincide with individual I by a clockwise rotation through 90°.

The two horizontal 4-fold axes of the pseudo-cube, [110] and [$1\bar{1}0$], are symmetrical to each other in the monoclinic mirror plane. In such a case it is customary to consider only one of the two, say [110], as a twin axis (that the other can also be a twin axis is a logical necessity, due to the symmetry of the crystal). Two twin laws arise, according as the angle of rotation is 90° (see observation No. 1) or 180° (see observation No. 2). Note, as above, that the 270° rotation does not yield a distinct twin law.

Two of the four 3-fold axes of the pseudo-cube, [021] and [$02\bar{1}$], are likewise symmetrical to each other in the monoclinic mirror. Only one, therefore, is considered, say [021]. Note that it is immaterial to use a 120° or a 240° rotation, as they lead to indistinguishable twins (see observation No. 3).

The other two 3-fold axes, [201] and [$\bar{2}01$], lie in the mirror plane of cryolite and are not equivalent. They give rise to two possible twin laws (see observations No. 12 and No. 13).

Of the two horizontal 2-fold axes of the pseudo-cube, [100] and [010], only [100] can be a twin axis (see observation No. 5); [010] coincides with the monoclinic 2-fold axis.

Of the remaining four 2-fold axes, only two should be considered, say [111] (see observation No. 10) and $[\bar{1}11]$ (see observation No. 4); the other two, $[1\bar{1}1]$ and $[\bar{1}\bar{1}1]$, are their mirror-images.

Of the mirrors perpendicular to the 4-fold axes of the pseudo-cube, only two need to be considered as possible twin planes, namely (001) (see observation No. 5) and (110) (see observation No. 9). The third one, $(1\bar{1}0)$, is the mirror-image of (110).

Of the mirrors perpendicular to the 2-fold axes of the pseudo-cube, one, namely (010), coincides with the monoclinic mirror and hence cannot be a twin plane. The following must be considered: (100), quasi-normal to [100] (see observation No. 6); (112), quasi-normal to [111] (see observation No. 7); and $(\bar{1}12)$, quasi-normal to $[\bar{1}11]$ (see observation No. 8).

The center of symmetry of the pseudo-cube coincides with the monoclinic center of symmetry, and hence cannot be a twin center.

The *index* and the *obliquity* have been calculated for the predicted twins (Table 1).

Observations on Record

The observations are found in Böggild (1912), to whose figures references are made in the following list.

(1) Twin axis [110], 90° rotation. Penetration twin; common. This is "Baumhauer's Law" (Figs. 4–5, Fig. 8). Note that common, penetration twins with twin axis $[1\bar{1}0]$, 90° rotation, have also been described (Fig. 6, Fig. 7). Such twins are properly included in Baumhauer's Law.

(2) Twin axis [110], 180° rotation. The rhombic section is here $(1\bar{1}0)$. The twin is of the repeated type; it is less common than the first two (Figs. 9–10, *et ss.*).

(3) Twin axis [021], 120° rotation. Irregular composition surface. This is Böggild's "new law";[1] it is common, especially in granular cryolite; it also occurs in fine lamellae; it is probably always secondary (Figs. 21–22, Fig. 23).

(4) Twin axis $[\bar{1}11]$, 180° rotation. The rhombic section is not a possible face, but it is close to (110). This twin is of the repeated type. It is rare; never obtained by gliding; never found in granular cryolite.

(5) Twin plane (001). The same law can also be defined by means of

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[1] In describing his new twin law, Böggild uses another definition, geometrically adequate, but which does not bring out the facts, (1) that a lattice row is the twin axis, (2) that the angle of rotation, in the twin operation, is of the form 360°/n, with $n=2, 3, 4,$ or 6. Böggild agrees that definition (3) is equivalent to his own.

the twin axis [100] with 180° rotation and composition plane (001). Found on granular cryolite only, in lamellae, common.

Table 1. Cryolite Twinning

Twin Law	Index	Obliquity
[001] 90°, 180°; (001)	1	0°11′
[110] 90°, 180°; (110)	2, 1, 1	1°58′
[201] 120°	2	0°47′
$[\bar{2}01]$ 120°	2	0°55′
[100] 180°; (100)	1	0°11′
[111] 180°; (112)	2	1°29′
$[\bar{1}11]$ 180°; $(\bar{1}12)$	2	1°18′

(6) Twin plane (100). The same law can also be defined by means of the twin axis [001], with 180° rotation and composition plane (100). Found on granular cryolite only, in lamellae, common.

(7) Twin and composition plane (112). Found on granular cryolite only, in lamellae, common.

(8) Twin and composition plane $(\bar{1}12)$. Found on granular cryolite only, in lamellae, common.

(9) Twin and composition plane (110). This twin occurs in lamellae and is found only on granular cryolite from the Urals.

(10) Twin axis [111], 180° rotation. The rhombic section is not a possible face, but it is near $(1\bar{1}0)$. This twin has not been found by Böggild, but it may possibly be Cross and Hildebrand's "law *d*" (1885).

(11) Twin axis [001], 90° rotation. This twin law is very close to (9). It is one of Paduroff's "new laws" (1925).

(12) Twin axis [201], 120° rotation. This twin law is very close to (8).

(13) Twin axis $[\bar{2}01]$, 120° rotation. This twin law is very close to (7).

Böggild did not attempt to differentiate (11) from (9), nor (12) from (8), nor (13) from (7).

It is interesting to note that, when Friedel wrote his memoir (1905), he listed only four known twin laws. They were: (5), (9), (6), and (8).

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TWINNING OF CRYOLITE*

Dorothy Wrinch, *Department of Physics, Smith College,*
Northhampton, Mass.

The immensely successful development of the atomic structure of minerals (Bragg, 1937) has made this subject a rich repository of exemplifications of structural principles. An attempt has long been under way to obtain clues to the structures of the native proteins from a study of their crystals (Wrinch, 1948). Even the comparatively few crystalline proteins which have been studied by classical or x-ray methods focus attention on two striking characteristics: a prevalence of crystals with high symmetry or pseudosymmetry, and a prevalence of twins and intergrowths. There can be little doubt that these two characteristics, possessed by few if any other types of organic materials, are highly significant. However, if any serious attempt is to be made to discover wherein this significance consists, a first line of enquiry is the answer to the parallel question for the many minerals (albeit a minority of all minerals so far investigated) which also share these characteristics.

Accordingly, the study of a number of such minerals is in progress and a general viewpoint is emerging. Minerals are selected whose atomic patterns are already known by x-ray structure analyses: in this way, we can test the viewpoint, step by step and see, without delay, whether it leads to useful conclusions. According to this viewpoint, the high symmetry or pseudosymmetry of certain crystals and the laws according to which they twin are regarded jointly as direct pointers to and direct indications of the nature of their crystal structure. On a previous occasion (Wrinch 1947), the viewpoint has been applied to staurolite and its pseudosymmetry and twinnings have been interpreted in terms of the (slightly disturbed) face-centered cubic oxygen network. From the standpoint of mineralogy, the point of interest was the way in which a classification of the twin laws of staurolite emerged to make, with the pseudosymmetry of the crystal, a simple and unified picture, directly indicating the nature of the atomic pattern.

The monoclinic mineral cryolite Na_3AlF_6, which is also pseudocubic, has long been a focus of attention for studies of twins (Friedel, 1905, 1926; Böggild, 1912); it exhibits at least 13 twin laws (Dana, 1951). It

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was therefore thought to be of interest to treat it as staurolite was treated, particularly as an x-ray structure analysis (Náray-Szabó and Sasvári, 1938) has yielded the crystal structure, though in it little interest is evinced in the pseudosymmetry or the twin laws.

MORPHOLOGICAL DATA

According to Krenner (Dana, 1892), cryolite is monoclinic ($2/m$) with $\beta = 90°11'$ and

$$a:b:c = 0.966:1:1.388.$$

Twin laws given in the new Dana (1951) based upon a statement by Donnay are as follows:

(1) By a 90° or 270° rotation on [110], penetration, common.
(2) By a 180° rotation on [110], rhombic section ($1\bar{1}0$), repeated, less common.
(3) By a 120° rotation on [021], composition surface irregular: common, especially in granular cryolite, as fine lamellae and probably always secondary.
(4) By a 180° rotation on [$\bar{1}11$], rhombic section near (110), repeated; rare, never found in granular cryolite.
(5) On (001) or by a 180° rotation on [100], composition plane (001).
(6) On (100) or by a 180° rotation on [001], composition plane (100).
(7) On (112), composition plane (112).
(8) On ($\bar{1}12$), composition plane ($\bar{1}12$).
(9) On (110), composition plane (110).
(10) By a 180° rotation on [111], rhombic section near ($1\bar{1}0$).
(11) On (211).
(12) By a 90° rotation on [001].
(13) By a 120° rotation on [201].
(14) By a 120° rotation on [$\bar{2}01$].

Study of the data

The description of the unit cell shows that it closely approaches and yet deviates measurably from a cell with β a right angle and axial ratio $\sqrt{2}:\sqrt{2}:2$, a cell which can have all its vertices on a simple cubic lattice with k as cube edge, provided that k is chosen to be in the range $a/\sqrt{2}$, $c/2$, $b/\sqrt{2}$. These facts suggest that the crystal, which is not cubic, may yet in some sense have a cubic character, a view of which is implicit in much of Böggild's great Memoir (1912). We then have to find out how this statement can be interpreted.

As before (Wrinch, 1947), we introduce this new cell, which may be called a compound cubic cell since all its vertices can lie at nodes of a single simple cubic lattice, as companion cell to the actual cell. We reformulate the coordinates xyz of the actual cell by introducing cubic coordinates $x_cy_cz_c$ which are fractions of k, as follows:

$$[100] = [110]_c, \qquad [010] = [\bar{1}10]_c, \qquad [001] = [002]_c;$$

so that

$$x_c = x - y, \quad y_c = x + y, \quad z_c = 2z; \qquad 2x = x_c + y_c, \quad 2y = y_c - x_c, \quad 2z = z_c.$$

We may then reformulate xyz planes in the cubic system $x_c y_c z_c$ and vice versa thus:

$$(200) = (110)_c, \qquad (020) = (\bar{1}10)_c, \qquad (002) = (001)_c,$$
$$(100)_c = (1\bar{1}0), \qquad (010)_c = (110), \qquad (001)_c = (002)$$

Here the first relation expresses the fact that the plane $x = 1$ is also the plane $x_c + y_c = 2$, and so on. This companion cell yields a multiple lattice of the k cube lattice, to which the actual lattice of the crystal is a close approximation, since k has been chosen appropriately. Its volume is $4k^3$. We remark that the $(2/m)$ symmetry of the actual crystal is now associated with the cube face diagonal $[\bar{1}10]$.

Proceeding as before we study the formulation, in the $x_c y_c z_c$ system of the companion cell, of the cubic forms of its embedded cubes, (x) denoting $(100)_c$, (s) and (t) denoting $(110)_c$ and $(111)_c$ and $[x]$, $[s]$ and $[t]$ denoting the normals to the planes; in terms of these planes and lines all the symmetry elements of the cubic system can be expressed.

$$(x) \equiv (002)_c, \qquad (200)_c, \qquad (020)_c,$$
$$= (001), \qquad (2\bar{2}0), \qquad (220)$$

yielding two forms, the pinacoid (001) and the rhombic prism (110).

$$(s) \equiv (110)_c, \quad (\bar{1}10)_c, \quad (011)_c, \quad (101)_c, \quad (0\bar{1}1)_c, \quad (\bar{1}01)_c,$$
$$(200), \quad (020), \quad (112), \quad (1\bar{1}2), \quad (\bar{1}\bar{1}2), \quad (\bar{1}12),$$

yielding 4 forms, two pinacoids (100) and (010) and two rhombic prisms (112) and $(\bar{1}12)$.

$$(t) \equiv (111)_c, \quad (11\bar{1})_c, \quad (\bar{1}11)_c, \quad (1\bar{1}1)_c,$$
$$(202), \quad (20\bar{2}), \quad (022), \quad (0\bar{2}2),$$

yielding 3 forms, two pinacoids, (101) and $(10\bar{1})$ and one rhombic prism (011).

We may proceed in the same manner with the directions normal to the planes of the various forms, putting together the axes which are symmetric for the crystal:

$$[x] \equiv [002]_c, \quad [200]_c \text{ and } [020]_c,$$
$$= [001], \quad [1\bar{1}0] \text{ and } [110],$$
$$[s] \equiv [110]_c, \quad [\bar{1}10]_c, \quad [022]_c \text{ and } [202]_c, \quad [02\bar{2}]_c \text{ and } [20\bar{2}]_c,$$
$$= [100], \quad [010], \quad [111] \text{ and } [1\bar{1}1], \quad [11\bar{1}] \text{ and } [1\bar{1}\bar{1}],$$
$$[t] \equiv [111]_c, \quad [11\bar{1}]_c, \quad [\bar{1}11]_c \text{ and } [1\bar{1}1]_c,$$
$$= [201], \quad [20\bar{1}], \quad [021] \text{ and } [0\bar{2}1].$$

In these results we observe the characteristic way in which single cubic forms correspond, for our compound cubic cell which does not have cubic symmetry, to two or more separate forms (Wrinch, 1947). If we refer to the forms observed for cryolite crystals (Dana, 1951), we remark the occurrence of all these forms.

Let us now examine the twin laws recorded, for any support which they can afford to the suggestion of some cubic character in the non-cubic crystal. This may be done, in a routine manner, by reformulating the planes and the axes occurring in the twin laws in terms of the $x_c y_c z_c$ coordinates of the companion cell. We may subdivide the twin laws, now in the two formulations, into a series of subsets:

A. Rotations of 180° and of 90° about the following axes:
  - Laws (6) and (12) $[001]=[001]_c$,
  - Laws (2) and (1) $[110]=[100]_c$ and $[010]_c$.

B. Rotations of 180° only about the following axes:
  - Law (5) $[100]=[110]_c$,
  - Law (10) $[111]=[011]_c$ and $[101]_c$,
  - Law (4) $[\bar{1}11]=[\bar{1}01]_c$ and $[0\bar{1}1]_c$.

C. Rotations of 120° about the following axes:
  - Law (13) $[201]=[111]_c$,
  - Law (14) $[\bar{2}01]=[\bar{1}\bar{1}1]_c$,
  - Law (3) $[021]=[\bar{1}11]_c$ and $[1\bar{1}1]_c$.

D. Mirrors in the following planes:
  - Law (5) $(001)=(001)_c$.
  - Law (9) $(110)=(010)_c$ and $(100)_c$,
  - Law (6) $(100)=(110)_c$,
  - Law (7) $(112)=(011)_c$ and $(101)_c$,
  - Law (8) $(\bar{1}12)=(\bar{1}01)_c$ and $(0\bar{1}1)_c$,
  - Law (11) $(211)=(131)_c$ and $(311)_c$.

We notice in this series of twin laws some remarkable features. The symmetry elements in A comprise the $\frac{1}{2}x$ and $\frac{1}{4}x$ rotations of the symmetry O_h. Those in B comprise correspondingly the $\frac{1}{2}s$ rotations of the symmetry O_h, except for the 180° rotation about the s line $[\bar{1}10]$, which is a symmetry element of the crystal itself. The symmetry elements in C, we notice, comprise the four $\frac{1}{3}t$ rotations of O_h. Thus all the rotational symmetry elements of O_h are now accounted for. The interpretation of the mirror twins is equally interesting. The crystal itself has an s mirror, the mirror in the s plane $(\bar{1}10)$. In virtue of this fact, it follows that one of the $\frac{1}{2}x$ symmetry elements, $\frac{1}{2}[001]$, is equivalent to an s mirror, $(110)_m$, that one of the $\frac{1}{2}s$ symmetry elements, $\frac{1}{2}[110]$, is equivalent to an x mirror, $(001)_m$. We duly observe these alternative formulations of law (6) and of law (5) in the subset D. In this subset we further remark the presence of the remaining x mirror, in law (9) and of two other s mirrors which, with the s mirror belonging to the crystal, complete all the symmetry elements involving mirrors belonging to O_h. The crystal itself has a center of symmetry. Already all the twin laws except (11) have been discussed. As we have, in fact, demonstrated in studying the twin laws, there are 13 and only 13 independent symmetry elements which, for our monoclinic crystal of symmetry $(2/m)$ associated with a cube face diagonal, are required to complete the symmetry O_h. The remarkable fact has now emerged that the 13 twin laws on the list, excluding law (11), provide just these symmetry elements. In a clear and useful sense, we may call the twins covered by these 13 laws "cubic" twins and the result may be expressed in the statement that, for the cryolite crystal, all possible "cubic" twins have been recorded. Only the twin law (11) lies outside this scheme.

There are two consequences of this analysis, one a matter of description, the other a question of interpretation. On the one hand we can now give a description of 13 of the twin laws, which shows that they constitute a coherent system, with no missing members: the 13 symmetry elements are those which, combined with the 2 symmetry elements of the crystal, give the symmetry O_h. That the 13 twin laws, for which no interrelations are suggested in the literature, can be so described is a fact of some interest, in and for itself. On the other hand, with the present viewpoint, an interpretation of this striking fact is readily forthcoming. The analysis was undertaken in response to the question of what support, if any, the twin laws can give to the hypothesis of some cubic character in the crystal. The answer is that there is complete support for the hypothesis, since every "cubic" twin law which can exist has been shown to occur.

If these striking facts have any significance, it must reside in the positions in the unit cell of the crystal of the known complement of $3\text{Na}+\text{Al}+6\text{F}$ ions or some multiple thereof. In the case of staurolite, sites for complements of atoms on nodes of cubic lattices associated with the companion cell were immediately suggested, and it was recognized that assigning the atoms to positions at or associated with such sites, such sites being subsequently transferred to the actual cell, would give a ready interpretation of the cubic quality of the crystal.

Proceeding after the same manner for the cryolite crystal, we remark that there are only 4 nodes of the simple cubic lattice of metric k in the companion cell to contrast with 10 ions in the elementary composite of cryolite. This k lattice is, of course, merely the coarsest of the possible cubic lattices on which the companion cell can place all its vertices and it is evident that it is too coarse for our purpose. We therefore proceed one step further, to cubic lattices depending on a $\frac{1}{2}k$ metric. Introducing the $\frac{1}{2}k$ simple-cubic lattice, we should get 32 nodes: there are also the intermediate cases, of a body-centered cubic lattice with 8 nodes and a face-centered cubic lattice with 16, all the nodes lying on the $\frac{1}{2}k$ lattice.

Actually the three types of cubic lattice involve superpositions. Thus, begin with 4 points of the k-lattice and supplement with the 8 points and the 16 points and there emerge only 20 points. These may be described as the nodes of the body-centered cubic lattice (at vertices and body-centers of the k cubes) plus only 12 nodes of the face centered cubic lattice: these 12 lie either at face-centers of k cubes (the vertices being already tenanted) or at mid-edges of k cubes (the body-centers being already tenanted). Furthermore, we may, if we wish, subdivide the body centered cubic lattice into two simple cubic lattices, with nodes at vertices of k-cubes and at body-centers of these cubes. Thus there are alternative formulations

$$20 = 8 + 12, \qquad 20 = 4 + 4 + 12.$$

The comparison with the composition of cryolite involving 10 ions is now very interesting. The first hypothesis for consideration is that there are two equivalents in the unit cell, with the 12F ions in positions associated with the 12 nodes of the (incomplete) face-centered cubic lattice in one or other of its expressions: there then remain, for 4Na ions positions associated with one or other of the simple cubic lattices, for the remaining 2Na+2Al positions associated with the nodes of the other. It is to be remarked that the subdivision among different ions of sets of nodes which form single equivalent sets for the cubic system is to be regarded, on the present viewpoint, as the meaning of the subdivision of a single cubic crystal form for the companion cell into two or more monoclinic forms of the actual crystal.

It is to be emphasized that the hypothesis just formulated is to be regarded simply as the first hypothesis to consider. Other hypotheses may be formulated, but only if still finer lattices involving smaller metrics than $\frac{1}{2}k$ are considered. Thus it would be possible to put in order a whole set of such hypotheses, each being of interest only if the preceding hypotheses have been shown to be untenable. One example may be given. In scrutinizing the composition of cryolite, the possibility of a face centered cubic lattice for the 3Na and Al ions early presents itself for consideration. But we readily see that this idea requires the introduction of lattices with a metric finer than $\frac{1}{2}k$. For giving the 3Na and Al ions positions associated with the nodes of even the coarsest face-centered cubic lattice in the cell, we are accommodating 4 such sets. There are then only 16 of the 32 positions with the $\frac{1}{2}k$ metric left, these are insufficient for the accompanying 24F ions. It is therefore necessary to proceed to a finer metric. Thus quite apart from any assessment of prior probability which may be given to this hypothesis for extraneous reasons, it is to be considered only if the preceding hypothesis involving only the $\frac{1}{2}k$ metric is discarded.

The actual crystal structure

We may now turn to the structure analysis which is given in the literature (Náray-Szabó and Sasvári, 1938). The findings are as follows:

Space group $P2_1/n$. $a=5.46$ Å, $b=5.61$ Å, $c=7.80$ Å, $\beta\sim 90°$. Cell contents $2(Na_3AlF_6)$

2Al	$000, \frac{1}{2}\frac{1}{2}\frac{1}{2}$;	4F	0.065, 0.06, 0.22, etc.
2Na	$00\frac{1}{2}, \frac{1}{2}\frac{1}{2}0$;	4F	0.71, 0.16, 0.03, etc.
4Na	0.50, 0.945, 0.24, etc.	4F	0.15, 0.28, 0.94, etc.

The hypothesis suggested is therefore correct in the number of equivalents per cell. Furthermore, the 2Na+2Al ions do indeed lie on one of the two simple cubic lattices—at vertices of *k* cubes. We should then, on our hypothesis, have the 4Na ions at the body centers of the *k* cubes, at $\frac{1}{2}0\frac{1}{4}$ etc., giving positions less than 0.4 Å from the positions actually found in the structure analysis. For the F positions, taking midedges of cubes, our hypothesis gives 4F at $00\frac{1}{4}$, etc., 4F at $\frac{\bar{1}}{4}\frac{1}{4}0$, etc., 4F at $\frac{1}{4}\frac{1}{4}0$, etc., which represent deviations from the actual positions which are about twice as great.

CONCLUSIONS

We may now assess the value of the approach to the crystal structure of cryolite which has been developed on the basis of the morphological data. The first of the hypotheses suggested, indeed the only hypothesis based upon the coarsest cubic lattice with sufficient sites to accommodate the 10 ions in the composition of the crystal, is essentially on the right lines. We see, in fact, that all the ions do indeed have positions associated with cubic sites. This means that, now in a precise sense, the statement can be made that cryolite, while not cubic, yet has a cubic character. It has also been shown how this interpretation of the cubic quality of this crystal affords a method of approach to possible sites with which the various ions of the crystal are associated. The part played by the twin laws in developing this argument perhaps needs emphasis. For most minerals, a twin law, like the twin law (11) in the present case, proves interpretable only after the crystal structure analysis has been completed. The remarkable feature in the present case is that there are 13 twin laws which are not only interpretable, as forming a coherent whole with the symmetry elements of the crystal, but serve as direct pointers to the nature of the crystal structure. Certainly the close approach of the shape of the unit cell to that of a compound cubic cell is very suggestive: however the set of 13 separate twin laws provide a much more detailed clue to the crystal. Together they constitute, as it were, the complete case which morphological data can present for a cubic quality in the crystal, with every symmetry element of O_h accounted for, either in the crystal or in the twin laws.

In scrutinizing the relations between the actual atomic positions in the unit cell of the crystal and the suggested cubic sites in the companion cells, two contrasting facts are worthy of comment. We remark the close approach of the positions of the 6Na and 2Al ions to points of a cubic space group—and the fact that positions associated with nodes of a *body-centered* cubic lattice might not have suggested themselves for these ions without the line of argument developed in this communication, makes it perhaps all the more remarkable—and we notice also the implications of this fact in relation to the symmetry elements of the 13 twin laws. But equally interesting is the extent of the deviation of the positions of the F ions from the nodes of the (incomplete) face-centered cubic lattice, with which, nevertheless, they can properly be said to be associated. Naturally we can make no estimate, *a priori*, as to the extent of the deviations of actual atomic positions from available cubic sites in a crystal of unknown structure in which both the shape of the unit cell and the nature of the twin laws combine to suggest some cubic character in the crystal structure. In the case of cryolite, we have the opportunity to study the inescapable manner in which the cubic character of a non-cubic crystal can demonstrate its presence, even when the deviation for a majority of the atoms is of considerable magnitude, through the morphology of the crystal and the morphology of its twins.

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Manuscript received Sept. 24, 1951

REMARKS ON "TWINNING OF CRYOLITE" BY DOROTHY WRINCH

J. D. H. Donnay, *The Johns Hopkins University*

AND

Gabrielle Donnay, *Geophysical Laboratory, Washington, D. C.*

Abstract

By selecting a sub-multiple cell, rather than a multiple cell, of the cryolite lattice to define the twin lattice, Dr. Wrinch succeeds in giving the twin lattice a physical significance. The generalization of the validity of this observation will require numerous additional examples.

In the preceding paper Dr. Wrinch establishes relationships between the twinning of cryolite and the crystal structure of this mineral. It is her implied hope that she will be able to predict some structural features of a crystal from its observed twin laws, by relating pseudo-symmetry to twinning and twinning to structure.

She starts with a lattice based on morphological elements—axial ratios and angle β. The pseudo-symmetry of this lattice leads her to choose a "companion cell," which is identical in shape with the "double cell" used by J.D.H.D. The two cells under consideration differ only by a scale factor: the edges of the "companion cell" are equal to A/2, B/2, C/2; those of the "double cell" being A, B, C. Since it is the pseudo-symmetry—not the size—of the "companion cell" that is used to predict the twin laws. Dr. Wrinch's predictions are identical with those of J.D.H.D. No less, no more.

The transformation "Náray-Szabó to Wrinch" is $\frac{1}{2}\bar{\frac{1}{2}}0/\frac{1}{2}\frac{1}{2}0/00\frac{1}{2}$. It amounts to centering the C-face of the cryolite cell and halving its *c*-axis. This is precisely the transformation which G. Friedel (1905), on the strength of the Law of Bravais, applied to the Krenner elements to find the lattice of cryolite. He states that the lattice thus obtained may be not the true crystal lattice but rather the "material lattice" (*réseau matériel*), meaning thereby that its nodes are occupied by atoms.

As to the relations of twinning to structure, Dr. Wrinch looks for a physical interpretation of the "companion cell" in the known crystal structure of the mineral. She finds that Na^+ and Al^{+++} ions play pseudo-equivalent roles at the nodes of the pseudo-cubic twin lattice, which—quite unexpectedly—turns out to be body-centered. Will atoms that are restored by twinning, in all cases, have to occupy nodes of the twin lattice, as they do in cryolite? The answer to this question should be sought in the remark that a twin operation, according to definition, is essentially a point-group operation, so that no operation involving a translation can be a twin operation. Returning to cryolite, it is hard to see how one would have gone about making predictions, had the crystal structure been unknown.

The term "twin lattice" is here used to designate the lattice which pervades the whole twinned edifice and governs the repetition of part of the crystal structure. Its cell is defined by the vectors $\boldsymbol{A}$, $\boldsymbol{B}$, $\boldsymbol{C}$, in the first paper; it is defined by $\boldsymbol{A}/2$, $\boldsymbol{B}/2$, $\boldsymbol{C}/2$, in the second paper. In the case of Dr. Wrinch's treatment, the twin lattice acquires a physical meaning: it is the pseudo-cubic body-centered lattice, the nodes of which are occupied by Na^+ and Al^{+++} ions. It is remarkable that these should be the small ions. One could have expected the large ions instead, since it is the packing of large ions that usually controls the crystal structure.

There is no reason for the privileged position given by Dr. Wrinch to the "cubicity" of the crystal. In fact the twin lattice based on her submultiple cell need not be pseudo-cubic at all. Any twin lattice resulting from the pseudo-perpendicularity of a net and a row will do just as well for the purpose. Many crystalline species are known in which twins are controlled by a cell that approximates a rectangular parallelepiped.

In summary, the outstanding point of interest in Dr. Wrinch's paper is the physical meaning given the twin lattice. Whereas the twin lattice used to be considered as a multiple lattice of the crystal lattice, Dr. Wrinch now makes the crystal lattice a multiple of the twin lattice. Part of the atoms of the motif can then be repeated by the twin lattice. Is this remarkable discovery limited to a few isolated cases, or does it have general validity? Since it is not accompanied by any theoretical explanation, it will have to be established by numerous examples before it can be accepted as a law of observation.

Manuscript received Sept. 24, 1951

SAPPHIRINE OCCURRENCE OF CORTLANDT, NEW YORK

Gerald M. Friedman, *University of Cincinnati, Cincinnati, Ohio.*

Abstract

Some of the properties of sapphirine are discussed. The sapphirine of Cortlandt has two modes of occurrence. One occurrence is in emery close to quartz veins, where it was formed by replacement of the spinel which is abundant in the emery. It also occurs in the cordierite-sillimanite hornfels close to the border of the Cortlandt norite, where in the process of deuteric alteration of the norite, magnesium-iron and aluminum oxides passed into the hornfels where they reacted with the silica of the schist to form the sapphirine.

Introduction

Sapphirine is a rare mineral. From the literature it appears that only nine occurrences of sapphirine have so far been reported.[1] Of these one has been recorded in the United States, which is the occurrence described in this paper. A study of the properties and origin of the American sapphirine, therefore, merits particular interest.

The author is indebted to Mr. Walter Maguire for the analysis of the sapphirine-bearing rock, also to Mr. and Mrs. Palmiotto and Mr. Joe De Luca for permission and encouragement to study the rocks on their respective mining properties.

Location and Geological Setting

The sapphirine occurrence is in Cortlandt Township, Westchester County, New York, located to the south and southeast of the town of Peekskill, about 35 miles north of New York City. The mineral occurs in the emery and cordierite-sillimanite hornfels of Emery Hill, Montrose, and on the west, southwest and south slopes of Salt Hill. Samples containing the mineral were obtained from the De Luca quarry at Emery Hill, from an abandoned quarry in Dutch Road, Montrose, from the Kingston mine in Colabaugh Road and from the emery and cordierite-sillimanite hornfels of the western and southwestern part of Salt Hill.

The igneous rocks of Cortlandt Township include peridotites, pyroxenites, norites, and diorites. These are intrusive into Manhattan schist, a strongly foliated quartz-mica schist. Emery deposits occur in the norite, usually close to its border with the schist. The schist where it directly adjoins the norite was subjected to thermal metamorphism and converted into a cordierite-sillimanite hornfels. Sapphirine occurs in both the emery and hornfels.

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[1] Palache, C., Berman, H., and Frondel, C., System of mineralogy: 7 ed., **1**, 724–726, John Wiley & Sons, New York (1946).

The emery is essentially composed of spinel, titaniferous magnetite and corundum. The relative proportions of these minerals vary widely. Some samples are largely made up of spinel and magnetite, others contain a high percentage of corundum and only a little spinel. Abundant quartz veins cut the emery. Garnet, sillimanite, cordierite, and sapphirine formed reaction rims around the quartz veins.

The hornfels, largely made up of cordierite and sillimanite, also contains abundant magnetite. Sapphirine occurs in this rock where it is close to the border of the norite.

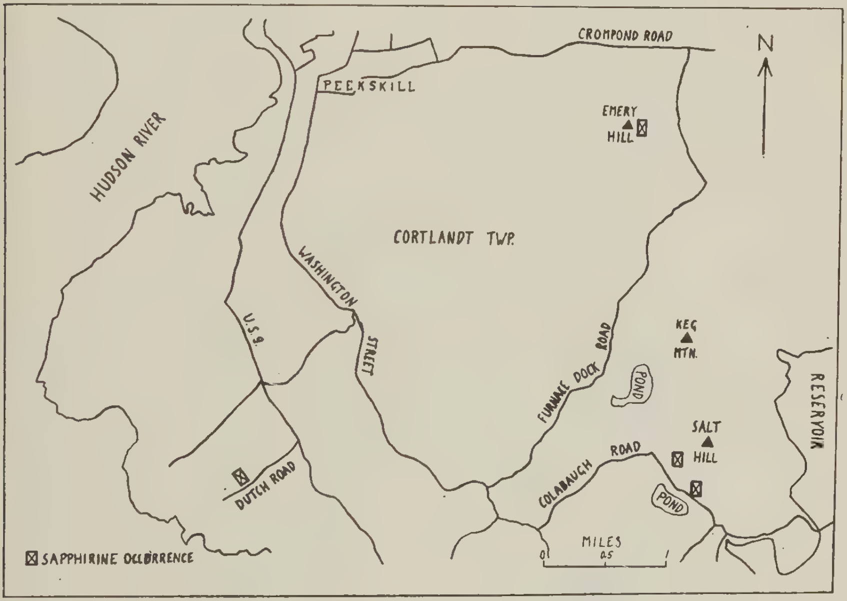

FIG. 1. Index map of Cortlandt Township, Westchester County, New York, showing location of sapphirine occurrences.

PROPERTIES

The sapphirine occurs in the form of small black grains. These vary widely in dimensions, the largest crystals range up to about 1.3 mm. in length and 0.5 to 0.7 mm. in width, the smallest ones are about 0.08 mm. in length and 0.04 mm. in width. Sapphirine crystals which are interspersed between sillimanite blades show extreme elongation, some of the crystals measured were 2.5 mm. in length and about 0.2 mm. and even less in width. Most of the sapphirine crystals are anhedral, some are euhedral to subhedral.

Many sapphirine crystals are studded with opaque inclusions, some of these are aligned to form parallel rods, others are unoriented. The mineral forming these inclusions appears to be magnetite.

Sapphirine is biaxial negative. Pleochroism is rather strong. α or X, yellowish-green, but varies slightly and may be pink; β or Y, light blue; γ or Z, moderate blue. Absorption $X<Y<Z$. Sections cut normal to *a* exhibit anomalous interference colors (grayish purple). Occasional crystals cut in other directions likewise show these colors.

Sections parallel to (100) show occasional polysynthetic twinning. {010} = twin plane; *b* is probably the twin axis. The presence of polysynthetic twinning in sapphirine has previously been reported from two foreign occurrences,[2] but details as to orientation were lacking. A possible second type of polysynthetic twinning with {100} as the twin plane was noted. Contact twins are abundant. This type of twinning has not been previously reported in sapphirine. Contact twinning is observable in plane polarized light because of the strong pleochroism of the mineral.

Cleavage parallel to {010} was noted but is not very distinct.

Occurrence in Emery

Sapphirine is not a primary mineral. It occurs only in emery that was cut by later quartz veins. Two modes of occurrence were noted. In one, the quartz veins are surrounded by reaction rims of garnet which in turn are followed by sillimanite layers which partly overlap the garnet; some sillimanite needles also occur in the quartz. The sapphirine crystals form an outer zone around the quartz veins and are interspersed with spinel grains. In the second type of occurrence, cordierite surrounds the quartz veins and is followed by sillimanite and sapphirine, the latter is in places interspersed with spinel grains.

Occurrence in Cordierite-Sillimanite Hornfels

The cordierite-sillimanite hornfels in Cortlandt Township is the "emery" of the quarrymen and the material that is commercially extracted. The most prominent occurrence of sapphirine in this rock is in the Kingston mine. The mine is bounded on the north by norite. Close to the norite wall the hornfels is largely composed of sillimanite, sapphirine, cordierite, and magnetite. Spinel relics occasionally abound. The sapphirine occurrence, however, is limited to a narrow zone close to the norite wall, beyond this zone sapphirine was not detected. Cordierite is more abundant, sillimanite and magnetite are less common at a greater distance from the norite wall. Spinel relics were noted only close to the norite border.

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[2] Palache, C., Berman, H., and Frondel, C., *op. cit.* (1946).

In the De Luca mine of Emery Hill sapphirine is likewise associated with sillimanite, magnetite, and cordierite. Spinel relics were also noted. The composition of the rock in which the sapphirine of Emery Hill occurs is as follows:[1]

	Per cent
SiO_2	16.35
Al_2O_3	53.12
Fe_2O_3	21.16
MgO	3.85
CaO	4.63
H_2O	0.46

Pittsburgh Testing Laboratory
Laboratory No. NY 73640

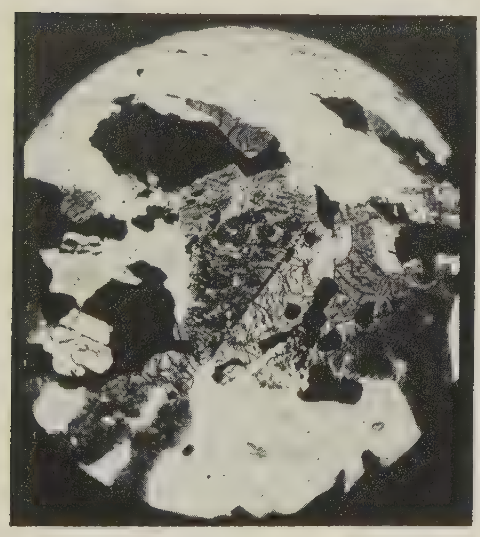

FIG. 2. Photomicrograph showing twins of sapphirine (centre of picture), untwinned sapphirine (at right and lower left), magnetite, cordierite (white), sillimanite (linear crystals in upper part of photograph), spinel (adjoining magnetite in upper part of photograph), and corundum (between magnetite grains in left). Plane polarized light. ×25.

ORIGIN

The sapphirine of Cortlandt Township is a replacement product of spinel. Spinel and quartz are incompatible. Where quartz veins cut emery a new suite of minerals formed resulting from the reactions between the spinel and quartz. These minerals include garnet, sillimanite, cordierite, and sapphirine. Quartz was nowhere observed to enclose or adjoin spinel directly, nor did spinel enclose the quartz without the presence of a reaction rim.

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[1] Walter Maguire, analyst.

The most conspicuous of the new suite of minerals is garnet which forms reaction rims up to $\frac{1}{2}$ inch thickness around quartz veins. Sillimanite likewise was formed in the reaction between spinel and quartz. It occurs as inclusions in quartz and garnet, forms an occasional zone between the garnet and spinel and is interstitial between the spinel grains. The following equation expresses the reaction involved:

$$\underset{\text{spinel}}{3(\mathrm{Mg, Fe})\mathrm{Al_2O_4}} + \underset{\text{quartz}}{5\mathrm{SiO_2}} = \underset{\text{garnet}}{(\mathrm{Mg, Fe})_3\mathrm{Al_2(SiO_4)_3}} + \underset{\text{sillimanite}}{2\mathrm{Al_2SiO_5}}.$$

Sapphirine likewise replaced spinel grains close to quartz veins and formed an outer zone around the latter. The sapphirine that replaced the spinel did not necessarily retain the latter's polygonal crystal pattern. Many sapphirine crystals replaced several spinel grains, thereby becoming somewhat elongated. Many spinel grains were only partially replaced. The reaction involved in the formation of sapphirine is represented by the following equation:

$$\underset{\text{spinel}}{15(\mathrm{Mg, Fe})(\mathrm{Al_2O_4})} + \underset{\text{corundum}}{2\mathrm{Al_2O_3}} + \underset{\text{quartz}}{7\mathrm{SiO_2}} = \underset{\text{sapphirine}}{(\mathrm{Mg, Fe})_{15}\mathrm{Al_{34}Si_7O_{80}}}.$$

Cordierite also formed reaction rims around quartz veins. If cordierite occurs, garnet is not normally noted, but sapphirine is closely associated with the cordierite. The formation of cordierite can be expressed as follows:

$$\underset{\text{spinel}}{2(\mathrm{Mg, Fe})\mathrm{Al_2O_4}} + 5\mathrm{SiO_2} = \underset{\text{cordierite}}{(\mathrm{Mg, Fe})_2\mathrm{Al_4Si_5O_{18}}}.$$

The formation of the sapphirine occurring in cordierite-sillimanite hornfels is in principle similar to that noted near quartz veins in emery. It appears, however, to be earlier than the sapphirine occurring in the latter. As demonstrated by Shand,[2] the pyroxenes and feldspars of the norite gave rise to poikilitic hornblende at a post-pyrogenetic stage of alteration. Magnesium, iron, and aluminum oxides were produced in the same process that gave rise to the hornblende. Shand being concerned with the formation of the poikilitic hornblende in the norite formulated the following equation:

$$\begin{aligned}&\underset{\text{diopside}}{\mathrm{Ca(Mg, Fe)Si_2O_6}} + \underset{\text{anorthite}}{\mathrm{CaAl_2Si_2O_8}} + \underset{\text{hypersthene}}{4(\mathrm{Mg, Fe})\mathrm{SiO_3}} + \mathrm{H_2O}\\ &= \underset{\text{hornblende}}{\mathrm{Ca_2(Mg, Fe, Al)_5H_2Si_8O_{24}}} + x\mathrm{Al_2O_3} + y(\mathrm{Mg, Fe})\mathrm{O}.\end{aligned}$$

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[2] Shand, S. J., Phase petrology in the Cortlandt complex, New York: *Bull. Geol. Soc. America*, **53**, 417–418 (1942).

Some of the magnesium-iron oxides gave rise to spinels and were formed concurrently with the hornblende. Most of the oxides, however, were transported by the solutions to the border of the norite where the emery was formed. Magnesium-iron oxides and to a limited extent aluminum oxides that passed into the adjacent schist reacted with the quartz of the schist to give rise to sapphirine close to the norite wall. The ratio of magnesium-iron oxides to silica in the sapphirine molecule is about 2:1. Near the norite border the concentration of the basic oxides was relatively high and sapphirine formed. At a greater distance from the norite the magnesium-iron oxides reacted with the silica of the schist to form cordierite in which the proportions of the basic oxides to silica are 1:5. A study of the chemical composition of the hornfels indicates a decreasing magnesium and iron, and an increasing silica content with increasing distance from the norite.

Sapphirine has been described from Guinea, Canada, Greenland, Italy, Transvaal, Madagascar, and India. In almost all of these localities the mineral association is similar to that of Cortlandt. Spinel is almost invariably associated with the sapphirine, and other emery minerals such as corundum and magnetite were cited from several of the occurrences. Pyroxenes, notably orthorhombic pyroxenes, prominent at Cortlandt, have been reported from most of the localities. Sillimanite and cordierite likewise occur. As the mineral assemblages of the sapphirine occurrences are very similar it seems likely that their modes of origin may also be related.

Manuscript received June 14, 1951

PRESENTATION OF THE MINERALOGICAL SOCIETY OF AMERICA AWARD TO ORVILLE FRANK TUTTLE*

NORMAN L. BOWEN, *Geophysical Laboratory, Washington, D. C.*

Mr. President, Fellows and Members of the Mineralogical Society of America, and guests:

This is a pleasant task.

The new award of the Mineralogical Society of America is, in my opinion, inspired. The Council which drew up the terms of the award must have had in mind the poet's query, "What are garlands and crowns to the brow that is wrinkled?" and his reply, " 'Tis but as a dead flower with May-dew besprinkled." In imposing a low age limit for eligibility the Council evidently felt that there were laurels aplenty to reward long records of achievement. There was needed an award to be given for a specific accomplishment indicating great promise, and to serve as a spur to the fulfillment of that promise by one still vigorous enough to respond. There must be no wasted May-dew. Above all there must be no mildew.

And in its decision as to the character of the award how wise the Council was! A life subscription to the *American Mineralogist* is not only valuable, but unlike most awards, it is very useful, and through it the recipient will feel anew the prick of the spur bimonthly through life. How well-designed it is to serve in the dual capacity of a reward and a spur!

As the first recipient the Council has chosen Orville Frank Tuttle, and the award is made for his work on the variable inversion of quartz. When Dr. Tuttle first mentioned to me that he was thinking of investigating the α-β inversion of quartz, I thought he was a bit dotty. The inversion of quartz had been investigated backwards and forwards and sidewise, and the Bureau of Standards had fixed it at $573.3 \pm 0.1°$ C. What more was there to learn about it? But Tuttle had a hunch, and the hunch paid off. He found that the inversion temperature was variable and was related in a systematic way to the manner of occurrence of the quartz, and consequently to the conditions under which the mineral formed, especially the temperature. Conversely, then, the variable inversion of quartz serves as a geologic thermometer. His results are a matter of record—indeed must be, in order to meet the conditions of the award. Jointly with Keith he has since expanded the investigation to cover natural and synthetic quartz formed under a wide range of conditions. The synthetic quartz is, of course, grown under measured conditions and therefore serves as a control. The new results are about ready for publication.

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* TUTTLE, DR. ORVILLE FRANK, Geophysical Laboratory, 2801 Upton St., Washington, D. C. GEOLOGY. Olean, N. Y., June 25, 16; m. 41; c. 2. B.S. Pa. State Col. 39; M.S. 40; Ph.D. (petrol), Mass. Inst. Tech., 48. Teaching fellow miner. and petrol., Mass. Inst. Tech, 40–41, 41–42, asst. Office Sci. Research & Develop., 42, physical chemist, Geophys. lab. 42–45; U. S. Naval Research Lab. Wash., D. C., 45–47; PETROLOGIST, GEOPHYS. LAB. 47–. Fel. Geol. Soc.; fel. Geophys. Union; Miner. Soc.; Geol. Soc. Wash. Phase equilibrium in silicate systems; rock forming minerals; structural petrology. (From: *American Men of Science*—Eighth Edition.)

Now as to future promise, which, I believe, is a major concern in this award. Tuttle has a flair for experiment that amounts to genius. He has a number of investigations under way, each as well chosen as his quartz investigation for its general significance in problems of mineral and rock genesis. The dew falls on a bud full of life and vigor, and ready to expand into a fine bloom.

Mr. President, I have the honor and pleasure of presenting Orville Frank Tuttle (may I say, in an aside, that if you wish to please him, call him Frank, not Orville, and conversely)—Orville Frank Tuttle for the first Mineralogical Society of America Award.

ACCEPTANCE OF THE MINERALOGICAL SOCIETY OF AMERICA AWARD

Orville Frank Tuttle, *Geophysical Laboratory, Washington, D. C.*

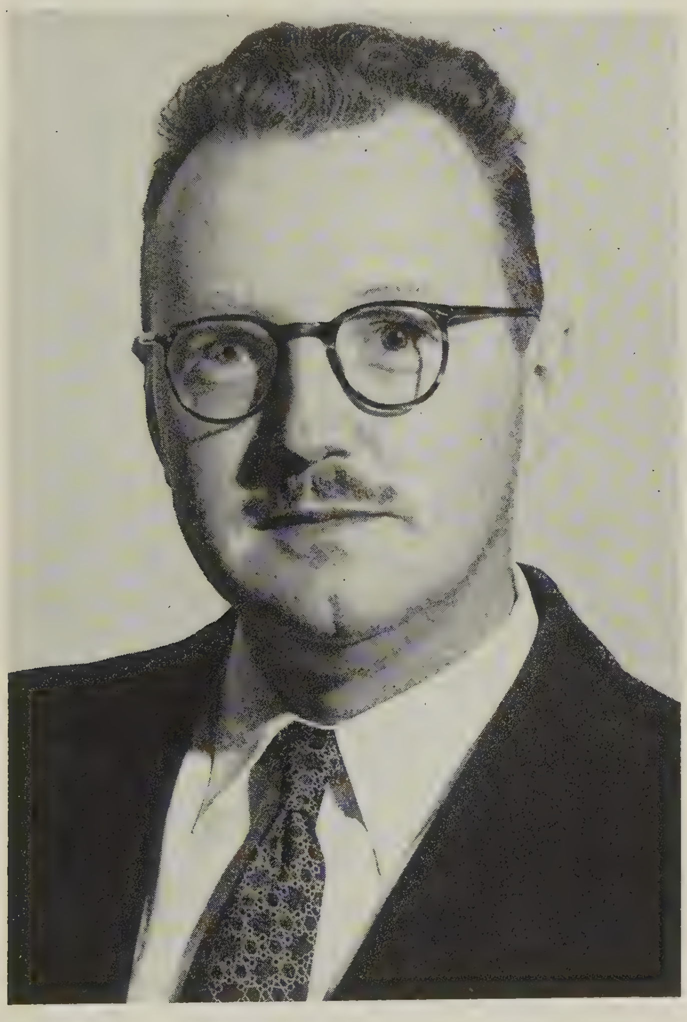
Mr. President, Dr. Bowen, Fellows and Members of the Mineralogical Society:

I am sure the Mineralogical Society Award has been conceived as an incentive to further research in the Mineralogical Sciences. I assure you that this purpose has been achieved. I am honored and greatly pleased by this award, but I hasten to say that I am well aware that my chances of fulfilling the requirements would have been slight indeed had I not been so fortunate as to have been associated with the Geophysical Laboratory during the past five years. Any contribution that I may have made to Mineralogy can be directly attributed to the encouragement and assistance of my co-workers at the Laboratory.

It seems appropriate to interject a few words concerning the present status of the quartz inversion studies despite the threats and warnings accompanying numerous suggestions to be brief in accepting this award. Dr. M. L. Keith and I are continuing the investigations. We have examined over 300 specimens of quartz from many types of occurrences. Quartz samples have been found which show no inversion, others with unusually strong heat effects and some with multiple inversions. Quartz from granites has, in general, different inversion characteristics from rhyolite quartz. Specimens of apparently homogeneous quartz from certain pegmatites may show several inversions and as many as three distinct quartzes could be isolated by careful hand picking under binoculars after heat treatment rendered them slightly different in color and lustre. We have synthesized quartz in special environments and by so doing altered the inversion temperature by as much as 40° C.

A manuscript is now being readied for publication bringing our results up to date and outlining plans for future studies. For obvious reasons, the quartz from granites and granites will receive high priority in the immediate future.

The complexities which are being discovered in the common rock-forming minerals have been the cause of some consternation. It seems to me that the fact that quartz can no longer be considered a constant of nature is no cause for alarm. On the contrary, the more complicated a mineral system becomes the greater its ability to store up information concerning its origin and subsequent history.



ORVILLE FRANK TUTTLE

Recipient of the Mineralogical Society of America Award

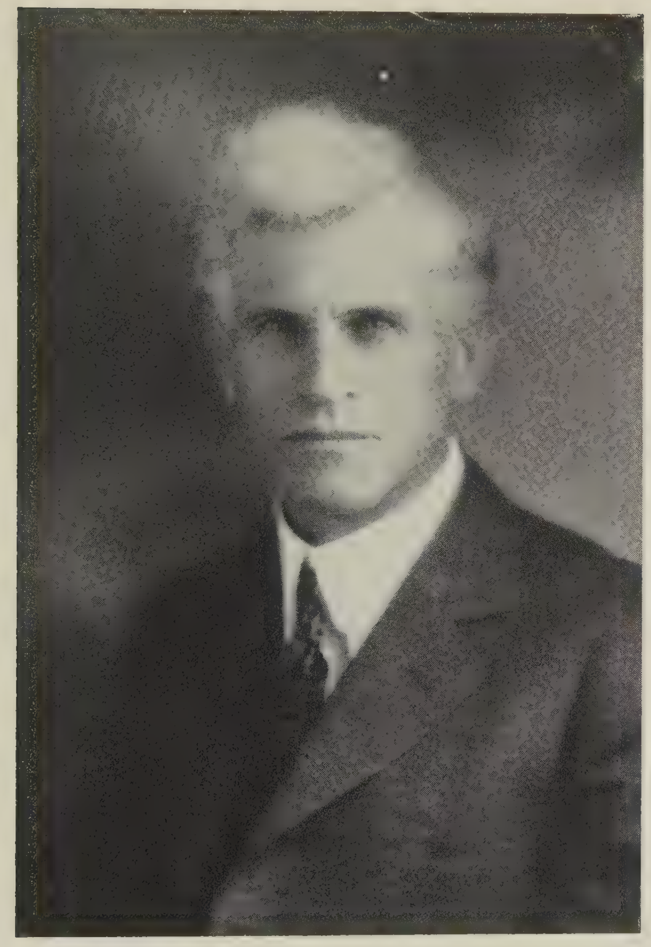
MEMORIAL OF RALPH DIXON CRAWFORD

Ernest E. Wahlstrom, *University of Colorado, Boulder, Colorado.*

Ralph Dixon Crawford, Emeritus Professor of Mineralogy and Petrography at the University of Colorado, passed away on March 7, 1950, the seventy-seventh anniversary of his birth. His death followed a surgical operation performed in an effort to prolong his life, and brought to an end the distinguished career of one of the ablest and most respected scholars of the Rocky Mountain region.

Professor Crawford was born in Peotone, Illinois, on March 7, 1873, and was next to the youngest of the ten children in the family of Ralph and Nancy Elizabeth (Cotes) Crawford. A younger brother, and older sister, and his wife, Theophania (Huntington) Crawford, survive him. He spent his youth in Illinois, and after receiving preparatory training at Northern Indiana Normal School (now Valparaiso University) entered the teaching profession and taught for several years in the public schools of Illinois, Texas, and Colorado. While in Colorado he became interested in geology and entered the University of Colorado, where he obtained a B.A. degree in 1905, and a M.A. degree in 1907. Upon completion of the work for the Master's degree he was appointed to an instructorship in Geology. Thus started a long and productive career of teaching and research at the University of Colorado. He interrupted his teaching to attend Yale University in 1910–11 and was awarded a Ph.D. by that institution in 1913, after having submitted a thesis on the Monarch-Tomichi District, Colorado.

Early in his career, Professor Crawford demonstrated considerable ability in the field study of geology and in the period from 1906 to 1925 spent much time in the mapping and interpretation of the geology of several Colorado areas. His first extensive field experience came when he served for two summers as field assistant to Hoyt S. Gale in a study of the coal fields of northwestern Colorado. During the next two summers he was assistant to the State Geologist of Colorado and worked in the Boulder County Tungsten District and the Hahn's Peak area of northwestern Colorado. In subsequent years he served as a geologist for the Colorado Geological Survey and was placed in active charge of several field and laboratory investigations. He supervised and actively participated in detailed geological studies of the Garfield, Monarch, Tomichi, Gold Brick, and Red Cliff districts of Colorado. In much of this work he developed a particular interest in igneous rocks and ore deposits, but his concise and authoritative reports covered all phases of the geology and mineralogy of the areas investigated by him and his co-workers.



Ralph Dixon Crawford
1873–1950

Although Professor Crawford established an enviable reputation as a research geologist and mineralogist, he is best remembered by his students as an inspiring and inspired teacher—a lovable person of unimpeachable honesty and dignity. He demanded and received the best that each of his students could give. He would not tolerate a slovenly performance and by his own actions set an example of high moral standards and intellectual endeavor for those who worked with him.

During the last twenty years of his life, poor health forced Professor Crawford to give up his field work and laboratory research, but in spite of his health, he was able to continue his duties as a teacher until 1940, when he retired as Professor Emeritus. During the many years that he taught, and after his retirement, he kept up an extensive correspondence with many former students, and by kindly interest and wise counsel made his influence felt long after they had graduated from college. Students and friends returning to Boulder always made a special effort to spend some time visiting with "Doc" Crawford in the pleasant surroundings of his home.

Professor Crawford's interest in people was almost matched by his keen appreciation of the significance of world events. He was a staunch Republican and spent many hours in animated discussions on political subjects. He was active in the Congregational Church and participated in many religious and charitable works. Scientific recognition was accorded him when he was elected a Fellow in both the Mineralogical Society of America and the Geological Society of America. He was also a member of the American Association for the Advancement of Science and Sigma Xi.

His character and personality were ably described by a pastor of his church who once said, "Doctor Crawford is one of God's own noblemen."

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Memorial received Feb. 15, 1951

MEMORIAL OF WILLIAM THOMAS GORDON

W. Campbell Smith, *British Museum* (*Natural History*), *London*

William Thomas Gordon, Professor Emeritus of Geology at King's College in the University of London, died in London on 13th December, 1950.

Much of his research work and nearly all his writings were on palaeontological subjects and particularly palaeobotany, but he had a considerable knowledge of mineralogy and he had made a good private collection, particularly rich in diamonds and precious stones, both cut and in the rough.

Gordon was born in Glasgow, 27th January, 1884, but his parents moved later to Edinburgh and he went to George Heriot's School there and later to the University, where he studied geology under James Geikie. He remained at Edinburgh as a research scholar after his graduation. Later he went to Cambridge as a research student for two years returning in 1912 again to Edinburgh as Lecturer in Palaeontology and Assistant in the Geology Department.

Just before World War I, Gordon was appointed Lecturer in Geology at King's College, London. The department at King's College was small and Gordon was the only teacher in it in 1914. After the war, he was made a University Reader in 1919 and Professor in 1920. By his energy and enthusiasm he soon built up a sound department and attracted many students to his courses. Soon after taking up his work in London, Gordon began to take an active interest in diamonds and other gem stones, influenced partly no doubt by the proximity of the main centre of diamond and gem stone trading in Hatton Garden to his own College in the Strand. He did much to assist the trade in the testing of rare stones.

He was a keen collector in the field himself and during his Edinburgh days, before he went to Cambridge, he had visited many of the Scottish mineral localities and by vigorous trenching of old dumps he had found many fine specimens.

On field excursions he was a delightful companion and to many visitors to London he was a cheerful and hospitable host. Many kindly and generous things were done by him to help others.

Outstanding among Gordon's numerous activities in London University was his devotion to the students and their social life. He played a large part in this side of the work of King's College. He was Senior Treasurer of the University Union, Warden of the College Hall of Residence, and President of the Old Students' Association.

William Thomas Gordon
1884–1950

His travels abroad were taken either to attend meetings of the International Geological Congress or of the British Association, or to take part in geological excursions with his students or the Geologists' Association.

He was President of the Geology Section of the British Association in 1934, a secretary of the Geological Society of London (1929–1937), Vice-President (1937–1939). He was a member of the Mineralogical Society of Great Britain (1923–1950), and of the Mineralogical Society of America (1927–1938) and a Fellow from 1938.

His mineralogical papers are very few and are mentioned below. An account of his geological and palaeontological work has already appeared in the obituary notice in the *Quarterly Journal of the Geological Society of London* (1951, **106,** Proceedings p. lx).

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Memorial received Jan. 21, 1952

MEMORIAL OF WILLIAM MARSH MYERS

S. Zerfoss, *Washington, D. C.*

Dr. William Marsh Myers, Chief of the Division of Mineral Economics in the School of Mineral Industries at the Pennsylvania State College died on January 25, 1951. The shock of his sudden death was shared with his family by his many friends and students.

The career of "Bill" Myers was comprised of a peculiarly happy blending of activity in the fields of science, engineering, economics, and education. His career in science was initiated when he took his B.S. degree at Syracuse University in 1914. This was followed by some graduate work at Columbia University in 1932. He received his M.S. degree in 1923 at the University of Michigan followed by a Ph.D. degree there in 1933 with a thesis entitled "Geology and Economics of the Exploitation of the Copper Ores."

He was employed as a chemist and metallurgist by the Crucible Steel Company (1914–1915) and by the Kalem Company, Inc. (1915–1917). His career as a mining engineer and geologist began with a year in the Peruvian copper mines of the Cerro de Pasco Copper Corporation (1917–1918) and continued with a year at the New Cornelia Copper Corporation Mine at Ajo, Arizona (1918–1919), and a year at the Homestead Iron Dyke Mine (1920–1921). The year at the "New Cornelia" was during the leaching period of that mine. Many and fascinating were the stories, both professional and otherwise, which have been related from this Arizona desert experience.

This practical experience as mining geologist was followed by six years (1921–1927) with the U. S. Bureau of Mines as Associate Mineral Technologist. One year of this period was spent in Washington, D. C., one year at Pittsburgh, Pennsylvania, and the remainder of the time at the New Brunswick station. The bibliography of his publications for this period shows his interest in mineralogy and mineral technology.

With this background in science and mining technology the next phase of his career was marked by an absorption of the economic aspect of the subject. He was service engineer for the U. S. Gypsum Company (1927–1932), thereby gaining valuable experience in the marketing of industrial minerals. Later he was associated with H. B. Earhart, Inc., of Detroit, Michigan, (1934–1938). He continued his activity as a mine evaluator until his death.

In 1938 Dr. Myers joined the staff of the School of Mineral Industries at the Pennsylvania State College as Assistant Professor of Mineral Economics and Technology. He became Associate Professor in 1942 and

William Marsh Myers
1892–1951

head of the new division of Mineral Economics in 1946. He was promoted to Professor of Mineral Economics in 1949.

No matter who has priority for the term "Mineral Economics" Dr. Myers made this particular bit of collegiate curriculum taxonomy peculiarly his own, both at school and in the mining society. Apart from his teaching, his formal contribution to mineral industries education was the formulation of a curriculum in Mineral Economics. In this he reflected his own experience by providing the student with a balanced diet of science, engineering, and economics. He was especially active in promoting this subject throughout Pennsylvania by papers, speeches and personal contact with the government at Harrisburg.

Dr. Myers carried his campaign for the recognition of this field of mineral industries into the A.I.M.E. and was a member of the first executive committee of the Mineral Economics Division of this Society.

This formal educational contribution was more than matched by the informal role as teacher of students. In the everyday classroom and in his office his students were much endeared to "Bill" Myers. The ideal instructor is one who can impart his enthusiasm for the subject to the students. Dr. Myers with his rapid-fire delivery of facts, opinions, and experience rarely failed to make his point on some mineralogical or economic problem. His intellectual curiosity was well evidenced in his classroom and private discussions.

Outside the classroom he was always available to students and friends for discussion, advice, and at times more substantial help. Many of us shared with him his hobbies too numerous to mention including his woodcarving and his interest in the violin.

Dr. Myers was a member of the A.I.M.E., a Fellow of the Mineralogical Society of America, and a member of Sigma Xi. He was a lifelong member of the Society of Friends.

Dr. Myers was born in Clinton, New York on November 22, 1892. On October 10, 1925 he married Ramona E. Reed. He is survived by his widow and his son William Bingham Myers.

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Memorial received Oct. 6, 1951

MEMORIAL OF HEINRICH RIES

Alfred L. Anderson, *Cornell University, Ithaca, New York.*

The sudden and unexpected passing of Dr. Heinrich Ries, Professor Emeritus of Geology of Cornell University, at his home in Ithaca, New York, on April 11, 1951, came as a shock to his many friends here and abroad. Death came in the midst of many activities less than three weeks before his eightieth birthday, and only two days before he was to have been the honored guest of the Central New York Chapter of the American Foundrymen's Association at a "Dr. Heinrich Ries Night." On that occasion he was to have been presented with a bronze plaque bearing the inscription, "In sincere appreciation of active leadership and contribution to the foundry industry in the field of sand research." His passing ended a long distinguished career as a teacher and mineral scientist in which he gained recognition as one of the leaders in the field of the nonmetallic mineral deposits. His pioneering work on clays and foundry sands will long be remembered and will remain a lasting monument of his achievements.

Dr. Ries was born in Brooklyn, New York, on April 30, 1871, the son of Heinrich and Caroline Bowman (Atkins) Ries. As a youth he had the advantages of an education obtained in part in this country and in part abroad. It is possible that his early studies and travels in Europe had served to arouse an interest in minerals, but in any event this interest was manifest when he enrolled as a student in the Columbia University School of Mines. He remained there to receive his Ph.B. degree in 1892 and then transferred to the School of Pure Science from which he was awarded an A.M. degree in 1894 and the Ph.D. in 1896. This training he supplemented with study at the University of Berlin and Polytechnikum during the winter of 1897–1898.

His long career as a teacher began while he was still a student at Columbia University. He served as Assistant in Mineralogy during the school year 1896–1897 and also as Lecturer in the Public Schools of New York City during the winters of 1895–1897. In 1898 Dr. Ries came to Cornell University as an Instructor in Economic Geology. In 1902 he was advanced to Assistant Professor and in 1906 to Professor of Economic Geology, a position he held until his retirement as Emeritus Professor in 1939. In 1914 he became the Head of the Department of Geology and served in that capacity for the next 23 years.

His professional career began while he was still at school and continued long after his retirement from academic work. He began his field work as an Assistant on the New York Geological Survey in 1891 and then

Heinrich Ries
1871–1951

served again in 1892 and 1895. His work in the field won him early recognition and after graduation from the Columbia University School of Mines he was assigned to a report on the clays of New York state. As very little attention had hitherto been given to the study of clays, Dr. Ries had to formulate objectives and methods and so successful was he in this and so well received was his report that his services were soon in demand by other States, the Federal Government, and Canada. He was thus engaged by the North Carolina Geological Survey in the Fall of 1895, the Maryland Geological Survey in the spring of 1897, the Alabama Geological Survey in the summer of 1898, the Michigan Geological Survey in the summers of 1899 and 1906, the New Jersey

Geological Survey in the summers of 1900 and 1901, the University of Texas Mineral Survey in the summer of 1903, the Wisconsin Geological Survey in the summer of 1904, the Virginia Geological Survey in the summers of 1905, 1916, and 1922, the Canada Geological Survey in the summers of 1909, 1910, 1911, 1912, and 1913, and the Kentucky Geological Survey in the summer of 1923. He was also employed as Special Agent, U. S. Geological Survey from 1895 to 1910 and again from 1918 to 1919. While busily engaged on the reports based on all this field work he also found time to write a book on clays, which also added greatly to his prestige.

Early in his career Dr. Ries became acquainted with Dr. Richard Moldenke, the celebrated metallurgist, and from this acquaintance developed an interest in foundry sands. Soon information on foundry sands was included in many of his State reports, and in the course of time more and more of his attention was directed to this important resource. When several technical societies organized a committee to study foundry sand back in the early twenties he was a member of that committee. From 1923 to 1928 he served as Chairman of the Committee on Standard Tests on Sands, American Foundrymen's Association, from 1928 to 1945 as Technical Director in charge of Sand Research, and from 1945 to his death as Chairman of the Association's Sand Division. At Cornell University he established the first laboratory in the country for research on foundry sands and much of the research that has been done on foundry sands since has been done in this laboratory. At the time of his death he was still active in directing this research. In recognition and appreciation of his work on foundry sands the American Foundrymen's Association awarded him the Joseph S. Seaman Gold Medal in 1936, and was prepared to honor him again with a testimonial dinner and bronze plaque on April 13, 1951, when death intervened. Honors also came from Alfred University which awarded him the Honorary Degree of Doctor of Science in 1945.

Recognizing the need of textbooks in the then young fields of Economic and Engineering Geology, Dr. Ries set to work to meet this need. In 1905 came the first edition of his well known and widely used text on *Economic Geology* which appeared in seven editions, the last in 1937. The text on *Engineering Geology* of which he was senior author had five editions. His book on *Clays, Occurrence, Properties, and Uses*, has appeared in three editions and was being readied for the fourth edition at the time of his death. Altogether he was author or coauthor of eight books prepared for use in the classroom.

Dr. Ries belonged to and took part in the activities of many professional and scientific societies. He was a Charter and Honorary Member of the American Ceramic Society and Honorary Member of the American Foundrymen's Association, the Rochester Academy of Science, and the Kentucky Academy of Science. In addition he had the distinction of Honorary Life Membership in the Canadian Institute of Mining and Metallurgy and the American Association for the Advancement of Science (Member in 1892, Fellow in 1898, and Life Member in 1939). He was a Life Fellow of the Geological Society of America and the American Geographical Society and a Life Member of the American Institute of Mining and Metallurgical Engineers. He was also a Member of the British Ceramic Society, the American Association of Petroleum Geologists, the Society of Economic Geologists, the Seismological Society of America, the Society of Economic Paleontologists and Mineralogists, and the National Geographic Society, and a Fellow of the Mineralogical Society of America. His activities in these organizations included a term as Vice President of the American Ceramic Society in 1903–1904 and President in 1910–1911. In 1925 he became Vice President of the Geological Society of America and in 1929, President. From 1902 to 1905 he was a Member of the Board of Managers of the American Mining Engineers and from 1910 to 1926, Chairman of the Committee on Non-metallics of the American Institute of Mining and Metallurgical Engineers. There were also the various appointments with the American Foundrymen's Association which occupied so much of his time in later years.

His activities were not entirely restricted to the affairs of the geological and other societies. In 1895 he served on the Jury of Awards at the Cotton States and International Exposition at Atlanta, Georgia; in 1901 the Pan-American Exposition in Buffalo, New York; and in 1904, the Louisiana Purchase Exposition in St. Louis, Missouri.

Dr. Ries took great delight in attending the meetings of the various societies to which he belonged. He missed very few meetings and could always be counted upon to be present at the annual meetings of the Geological Society of America and affiliated Societies and the annual meetings of the American Institute of Mining and Metallurgical Engineers. He delivered few addresses in later years but often entered into discussions. He preferred to spend his time in the halls where he could meet and converse with people. The papers could be read later and more profitably in the privacy of his study. Other meetings from which he obtained much enjoyment were those of the International Geological Congress. It was his good fortune to attend the meeting in Russia in 1897 as a delegate from Columbia University and the meetings in France in 1900, in Mexico in 1909, in Canada in 1913, and in Washington in 1933 as delegate from Cornell University.

Dr. Ries was a kindly person who enjoyed the company of other people. He was never too busy to put his work aside and listen to what others had to say. His office was always open to his students and off-campus friends and he was always ready to discuss their problems with them. He was deeply interested in his students and this interest did not end at graduation. It was his desire always to keep in touch with his graduates, watch their progress, and assist them in any way he could to better their condition. He maintained a voluminous correspondence not only with his students and former students but also with a host of others with whom he had become acquainted. He was always at home to those who wished to see him and encouraged his students to come to his home on Sunday afternoons.

His interest in his friends and people added greatly to his enjoyment of travel. In later years he kept his summers open so that he could be free to travel as he saw fit and do the things he enjoyed doing most. It gave him the greatest of pleasure to plan trips to the West Coast, trips which in the course of time became an annual event. He scheduled his trips so as to include summer meetings of the societies to which he belonged which gave him opportunity to meet more of his friends and make more acquaintances. Then accompanied by friends he enjoyed visiting places of geologic interest, and particularly those places which would afford him data for his lectures and for his books on economic and engineering geology. As expected, Dr. Ries was most interested in visiting the non-metalliferous deposits which throughout his life remained his chief concern. However, he had a deep appreciation of mountain scenery and the geomorphic processes behind the scenery and arranged to include trips to the National Parks and other places of scenic and geologic content. His friends in the West are going to miss these pleasant summer excursions.

Dr. Ries was a tireless and prodigious worker. Idleness was not a part of his life. When compelled to retire from academic work, he yielded gracefully but transferred his energies to his sand research and to committee and consulting work. He would have none of the armchair repose that is supposed to come to one after a long job well done. His interest was in the future and there was always the work of tomorrow. This work must now be carried on by the students whom he helped to train. We all regret the passing of one of the last of the older school of pioneering geologists. His contributions will long be remembered. He served long and faithfully and led an active and full life to the very day of his death.

His first wife, Mrs. Millie Timmerman Ries, died in 1942. He remarried in 1948, but his second wife, Mrs. Adelyn Halsy Gregg Ries, passed away early in 1950. He is survived by two two sons, Professor

Victor H. Ries of Ohio State University, and Professor Donald T. Ries of Illinois State Normal University.

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Memorial received Nov. 28, 1951

PROCEEDINGS OF THE THIRTY-SECOND ANNUAL MEETING OF THE MINERALOGICAL SOCIETY OF AMERICA AT DETROIT, MICHIGAN

C. S. Hurlbut, Jr., *Secretary.*

The thirty-second annual meeting of the Society was held on November 8–10, 1951, at the Hotel Statler, Detroit, Michigan. Scientific sessions were held on each afternoon and on the morning of November 10th, at which forty-nine papers were presented. In addition, on the morning of November 9th the Society, in collaboration with the Geological Society of America, sponsored a symposium on *Distribution of Igneous Rocks in Space and Time.*

The annual luncheon of the Society on November 8th was attended by 150 fellows, members, and guests. Following the luncheon the first presentation of the *Mineralogical Society of America Award* was made to O. F. Tuttle. The Society was then addressed by the Retiring President, A. Pabst, on *Mineralogical Notes.*

The increase in membership and fellowship in the Society has brought about corresponding increase in the time required by the Council to transact the business of the Society. The Council met for over ten hours during November 7th and 8th, and debated twenty-six items of business. The following are of particular interest to the general membership:

1. *Special Publication Series.* The Council voted that the Society sponsor a Special Publication Series comprised of papers of mineralogical interest that are too long to be printed in *The American Mineralogist.* The series is to be guided by an Editorial Board composed of the following:

F. A. Bannister
A. F. Buddington
M. Fleischer
C. Frondel (Chairman)
E. F. Osborn
A. Pabst
L. S. Ramsdell

2. *Suspension of Dues.* The Council voted to place the following resolution as an amendment to the By-laws on the ballot in 1952 to be voted upon by the general membership:

> Fellows and members who have reached the age of 70 years, or have reached the official retirement age of the institution in which they have been serving, and have paid annual dues for 30 years shall be exempt from further payment of dues but shall retain all rights and privileges.

3. *Editorial Board of The American Mineralogist.* The Council voted to appoint L. S. Ramsdell Assistant Editor of *The American Mineralogist* and to place the present Associate Editors on an Editorial Board without specific designation of fields.

The committee appointed by the Council to consider candidates for nomination to fellowship is given below. It is hoped that Fellows of the Society will suggest candidates to the committee members.

NOMINATING COMMITTEE FOR FELLOWS 1952

3 years—Herbert Insley (Chairman), and A. F. Buddington
2 years—Samuel Zerfoss and William Parrish
1 year—E. F. Osborn and George Faust

REPORT OF THE SECRETARY

To the Council of the Mineralogical Society of America:

Election of Officers and Fellows

Four hundred and thirty-four ballots were cast in the election of officers: 153 by fellows and 281 by members of the Society. The officers elected to serve in 1952 are:

President: Michael Fleischer, U. S. Geological Survey, Washington, D. C.
Vice-President: J. D. H. Donnay, The Johns Hopkins University, Baltimore, Maryland.
Secretary: C. S. Hurlbut, Jr., Harvard University, Cambridge, Massachusetts.
Treasurer: Earl Ingerson, U. S. Geological Survey, Washington, D. C.
Editor: Walter F. Hunt, University of Michigan, Ann Arbor, Michigan.
Councilor (1952–55): Victor T. Allen, Institute of Geophysical Technology, St. Louis, Missouri.

According to the provisions of the Constitution, the following have been elected to fellowship:

Carl W. Correns, Universität, Göttingen, Germany.
Robert Marx Dreyer, University of Kansas, Lawrence, Kansas.
Wilhelm Hermann Julius Eitel, Electrotechnical Laboratory, Norris, Tennessee.
Wilfrid Raymond Foster, Champion Spark Plug Company, Detroit, Michigan.
Robert Alchin Hatch, Electrotechnical Laboratory, Norris, Tennessee.
Richard Henry Jahns, California Institute of Technology, Pasadena, California.
Richard Childs Mielenz, Bureau of Reclamation, Denver, Colorado
Werner Nowacki, Universität, Bern, Switzerland.
Hans Seifert, Westf. Landesuniversität, Münster, Germany.
Hugo Strunz, Mineralogisch-Geologisches Institut, Regensburg, Germany.
Helmut G. F. Winkler, Universität, Göttingen, Germany.

MEMBERSHIP STATISTICS

November 1, 1951

	1950	*1951*	*Gain*	*Loss*
Correspondents	5	5	0	0
Fellows	307	309	9	7
Members	747	797	140	90
Subscribers	632	735	156	53
	1691	1846	305	150

The above figures show a net gain of 2 fellows, 50 members, and 103 subscribers. Considering the four groups together, there is a gain of 155, giving a total of 1846.

The Society lost through death four fellows: Lloyd Fisher of Bates College, Lewiston, Maine; William T. Gordon of Kings College, London, England; William M. Myers of Pennsylvania State College; and Heinrich Ries of Cornell University, Ithaca, New York.

Respectfully submitted,
C. S. Hurlbut, Jr., *Secretary*

REPORT OF THE TREASURER FOR 1951

To the Council of the Mineralogical Society of America:

Your treasurer submits herewith his annual report for the year beginning November 1, 1950 and ending October 31, 1951.

Receipts

Dues and subscriptions	$ 7,495.30
Sale of back numbers	1,298.49
Authors' charges on reprints	1,830.31
Interest and dividends from endowment	4,098.73
Geological Society of America aid for printing the Journal	7,006.19
Advertising	744.09
Sale of Index, volumes 21–30	104.10
Sale of Index, volumes 1–20	5.90
Bonds called	4,200.00
Contribution to Larsen issue (September–October 1950)	1,944.00
	$28,727.11
Cash on hand, November 1, 1950	7,172.06
	$35,899.17

Disbursements

Printing and distribution of the Journal (6 issues)	$14,942.57
Printing and distribution of reprints	1,972.08
To the Editor, Secretary and Treasurer	1,250.00
Clerical help	685.30
Postage and express	438.78
Printing and stationery	243.86
Office supplies and equipment	8.69
New securities purchased	9,678.67
Commission on securities	92.16
Taxes on securities	.30
Dividend adjustment on new stock	9.90
1950 program and abstracts	328.94
Expenses of officers to 1950 annual meeting	74.48
Expenses of committees	33.84
Safety deposit box	7.80
Back numbers purchased	27.70
Telephone and telegraph	4.04
Checks returned	8.00
Refunds	3.60
Exchange charges on checks	4.09
Contribution to America Geological Institute	400.00
Binding volumes of *American Mineralogist* for sale	82.50
	$30,297.30
Cash balance, October 31, 1951	5,601.87
	$35,899.17

The endowment funds of the Society as of October 31, 1951, consist of the following securities:

Bonds

6M Atlantic Coast Line, 4½	$5,257.50	
5M New York Central, 5	4,300.00	
5M Southern Railway, 5	5,743.75	
4C Great Northern, 5½	400.00	
		$15,701.25

Preferred Stocks

200 shares, Southern California Edison, 4.88	$5,250.00	
100 shares, Union Pacific, 4	4,570.25	
60 shares, Jones & Laughlin, A, 5	4,987.50	
55 shares, United States Steel, 7	6,946.20	
50 shares, Virginia Electric & Power Co., 5	5,942.50	
24 shares, Public Service Electric & Gas Co.	728.40	
10 shares, Consolidated Edison, 5	1,066.64	
		$29,491.49

Common Stocks

288 shares, Potomac Electric Power Co.	$3,871.00	
200 shares, Greyhound Corp.	2,300.00	
100 shares, Columbus and Southern Ohio Electric	2,087.50	
80 shares, Kroger Co.	1,990.00	
60 shares, United Fruit Co.	3,067.50	
56 shares, Standard Oil of New Jersey	1,444.84	
50 shares, Chesapeake & Ohio Railway	2,368.75	
50 shares, Pennsylvania Railroad	1,468.75	
47 shares, American Telephone & Telegraph	6,427.32	
40 shares, Plymouth Cord	2,050.00	
30 shares, U. S. Playing Card	2,411.25	
		$29,258.91
		$74,451.65

Respectfully submitted,
Earl Ingerson, *Treasurer*

For Dana Fund Report, see next page.

DANA FUND

Disbursements are made to needy mineralogists in war areas and to needy families of deceased mineralogists in war areas.

RECEIPTS

Available balance, November 1, 1950	$212.75
Interest	2.08
	$214.83

DISBURSEMENTS

Disbursed	$ 43.87
Available balance, October 31, 1951	170.96
	$214.83

Respectfully submitted,
EARL INGERSON, *Treasurer*

REPORT OF THE AUDITING COMMITTEE

To the President of the Mineralogical Society of America:

The Auditing Committee has examined and verified the accounts of the Treasurer of the Mineralogical Society of America for the fiscal year ending October 31, 1951. The securities listed in the Treasurer's report, with all future coupons on the coupon bonds attached, are in the safety deposit box at the West End Branch of the Washington Loan and Trust Company in Washington, D. C.

Respectfully submitted,
MARIE L. LINDBERG
THEODORE BOTINELLY
GEORGE SWITZER, *Chairman*

REPORT OF THE EDITOR FOR 1951

To the Council of the Mineralogical Society of America:

In presenting the annual editorial report at this time, the same policy will be followed as that used in recent years. With five issues published and distributed and only the November-December issue in press, sufficient data are now available to give a general survey of the year's activity. When the editor's report appears in print in the March-April number certain data incomplete or not available at present will be incorporated so that the final printed report in April can be compared with those given for previous years.

From the standpoint of articles published and total pagination the activity of 1951 can be considered that of a normal year. Aside from the regular issues, attention might be called to one so-called special number, authorized by the Council, and sponsored by the Walker's Mineralogical Club. This particular issue continued the series of Contributions to Canadian Mineralogy, and appeared as the May-June issue under the editorship of L. G. Berry of Kingston, Ontario. In this number the guest editor assembled 16 interesting papers on varied mineralogical subjects and assumed full responsibility in seeing these contributions through the press. This policy, followed for the past three years, of devoting one issue each year to Canadian mineralogy and Canadian authors seems to have won universal approval, and it is hoped that the Council will approve its continuance in 1952.

The current volume will run about 950 pages and contain 68 leading articles. Thirty-one additional short papers, appearing under Notes and News, give an over-all total of 99 published manuscripts for the year. As an indication of the general service rendered by the Journal in 1951, it may be mentioned in passing that 24 contributions were published from contributors residing outside of the States: 17 from Canada, and one each from Australia, Bolivia, Holland, Japan, New Zealand, South Africa, and Spain. These contributions were received from 113 authors associated with 50 different Universities, research bureaus, and technical laboratories. Twenty-six critical book reviews and 84 abstracts of new mineral names comprise other items of general mineralogical interest. Six new minerals were described in detail for the first time—andersonite, bayleyite, ferrocarpholite, novacekite, sabugalite, and swartzite.

While it is impossible at this early date to state with any degree of accuracy the printing demands that will be made on the Journal in 1952, it would seem that the volume will at least reach the size of that issued this year. This estimate is based on the fact that as of November 1 the files contain 36 articles of various lengths on a wide range of subjects aggregating about 485 typed pages. If, however, as has been tentatively suggested, a special number dedicated to a renowned mineralogist be included in the offerings, the total size may well exceed that of the current volume.

As a matter of permanent record, recognition and appreciation is here expressed for the valiant service performed by Dr. Michael Fleischer who for the past 11 years has had charge of the New Mineral Names section of the Journal. His critical remarks and good judgment in evaluating the propriety of proposed new mineral names has been of inestimable service. Also appreciation is expressed for the continued liberal support received from the Geological Society of America toward defraying a substantial portion of the printing costs. For this assistance the Society is indeed very grateful as otherwise dues would have to be increased or restrictive measures applied to keep the costs within our established income.

A suggestion has been received that if followed would slightly decrease the size and cost of the March issue, namely the elimination of the reprinting of the abstracts of papers read at the annual meeting. Since these abstracts are mailed out separately prior to the meeting, their reprinting as part of the printed Proceedings may not be necessary. However, the saving would not be large as the type is held over. The reprinting does permit corrections to be made as due to lack of time proof reading is not possible before the preliminary abstracts go to press. Also the reprinting makes it possible for the abstracts to become an integral part of a regular issue.

As a matter of convenience, starting with the January–February issue, each number will have a printed back bone carrying year, volume number and pagination. This bit of refinement should enable the reader to select more quickly the proper reference when the volumes are in a vertical position on the book shelves.

The accompanying Table 1 summarizes in detail the distribution of subject matter in volume 36.

TABLE 1. DISTRIBUTION OF SUBJECT MATTER IN VOLUME 36

Subjects	Articles	Pages	Per Cent of Total
Leading articles*			
Descriptive mineralogy	17		
Geochemistry	18		
Structural crystallography	14		
Petrography	8		
Geometrical crystallography	1		
Optical crystallography	5		
Memorials	2		
Miscellaneous	3		
	68	742	80.0
Shorter articles	31	77½	20.0
Notes and news	21	8½	
Proceedings of Societies	5	58½	
Book reviews	26	26½	
New mineral names	84	15	
Total entries	235	928	100.0
Illustrations	260		
Index, Title page, Table of contents		20	
Grand total		948	

* Leading articles average 10.9 printed pages each.

Respectfully submitted
WALTER F. HUNT, *Editor*

ABSTRACTS OF PAPERS PRESENTED AT THE THIRTY-SECOND ANNUAL MEETING OF THE MINERALOGICAL SOCIETY OF AMERICA AT DETROIT, MICHIGAN, NOV. 8–10, 1951

IONIC RADII OF THE ELEMENTS

L. H. AHRENS
Massachusetts Institute of Technology, Cambridge, Massachusetts

Of the two sets of ionic radii in common use—(1) Goldschmidt and (2) Pauling—those of Goldschmidt are usually employed by crystallographers, mineralogists and geochemists. Their use in preference to the other set is not supported by the relationship between ionic radius and ionization potential.

Within an isoelectronic sequence, ionic radii vary regularly with ionization potential. This is particularly well developed when Pauling radii are employed and is interpreted as strongly supporting their use in preference to those derived purely from measured interatomic distances. Other regularities between radius and ionization potential, and between radius and charge, are also well developed when Pauling radii are used.

Deviations by measured (Goldschmidt) radii can usually be explained by:

(1) an assumed radius of 1.32 A for O^{2-} as too low,

(2) significant polarization of O^{2-} by cations of high polarizing power, and

(3) some inaccuracies in interatomic distance measurements.

Radii in the isoelectronic sequence, Li^+-Be^{2+}-B^{3+}-C^{4+}-N^{5+}-O^{6+}-F^{7+} have been revised on a basis of a radius of 0.68 A for Li^+ rather than 0.60 A as used by Pauling.

Hitherto unknown radii for W^{6+}, Po^{6+}, Re^{7+}, Tc^{7+} and At^{7+} have been determined graphically by means of the well-developed regularities between ionization potential, charge and radius.

SALITE AND ACTINOLITE AT IRON MOUNTAIN, MISSOURI*

VICTOR T. ALLEN AND JOSEPH J. FAHEY
Saint Louis University, Saint Louis, Missouri;
U. S. Geological Survey, Washington, D. C.

Salite, actinolite, dolomite, and fluorite at Iron Mountain, Missouri, are reported for the first time. Salite, an intermediate pyroxene of the diopside-hedenbergite series, occurs in a skarn formed by the action of iron-bearing solutions on andesitic lava of pre-Cambrian age. The salite occurs as grayish-green columns up to 10 inches long; it has the following chemical composition and optical properties: SiO_2, 52.76; Al_2O_3, 1.12; TiO_2, 0.16; Fe_2O_3, 1.73; FeO, 8.92; MnO, 0.47; CaO, 20.48; MgO, 13.43; Na_2O, 0.35; K_2O, 0.05; H_2O, 0.45; $\gamma=1.715$; $\alpha=1.695$; $2V=60°$; positive; $Z\wedge c=45°$; density, 3.350. At somewhat lower temperature salite was locally changed to actinolite, which has previously been called amphibole or tremolite, but optical properties of numerous grains fall within the range of actinolite with $\gamma=1.645$; $\alpha=1.625$; $Z\wedge c=15°$. The iron garnet, andradite ($N=1.88$), replaces actinolite, and euhedral, zoned dodecahedrons contain relict fibers of the actinolite. Calcite and quartz replace actinolite and are cut by veins of hematite. Purple fluorite occurs in quartz, but is not in contact with hematite, so its age relation to hematite is unknown. A pink carbonate, having the color of rhodochrosite, occurs in veins cutting the andesitic lava, but it contains only 1.45 per cent MnO and has the optical properties of dolomite with $\omega=1.685$. Thus, doubt is raised concerning the rhodochrosite and manganocalcite reported in 1895 from this deposit.

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* Publication authorized by the Director, U. S. Geological Survey.

PHOSPHATE MINERALIZATION AT BOMI HILLS AND BAMBUTA, LIBERIA, WEST AFRICA

J. M. AXELROD, M. K. CARRON, C. MILTON AND T. P. THAYER
U. S. Geological Survey, Washington, D. C.

An interesting suite of iron phosphate minerals formed by biogeochemical processes has been found at Bomi Hills and at Bambuta in western Liberia. The phosphates at Bomi Hills form bouldery aprons on the slopes below two prominent cliffs and occur in caves and open fissures in cliffs of high-grade iron ore where they were formed by the interaction of bat dung, or substances derived from it, with exposed iron oxide. The substances thus formed include strengite and its dimorph, phosphosiderite; a member of the rockbridgeite group (formerly included with dufrenite); and most noteworthy of the suite, a complex substance, or possibly mixture of substances, apparently closely related to leucophosphite, described by Simpson (1932) from Western Australia.

The evidence, admittedly not conclusive, for regarding the Australian and African substances as constituting a mineral species is considered; their provisional acceptance as such rests on their similar and unique *x*-ray diffraction patterns, and essentially similar chemical composition—namely, potassium-ammonium-ferric hydrous phosphate. The paragenesis of the several phosphate minerals is discussed, based largely on petrographic study of the various specimens, with identification of the crystalline phases by *x*-ray diffraction patterns.

STUDIES OF METAMICT MINERALS (I): METHODS AND PROCEDURES*

JOSEPH BERMAN
U. S. Geological Survey, Washington, D. C.

Review of the literature reveals much confusion in the identification of metamict minerals and in interpretation of results obtained by *x*-ray powder studies. The need for standard methods of studying these minerals is urgent.

On the basis of *x*-ray powder patterns it is ascertained that many minerals containing radioactive elements show various degrees of "metamictization." Reproducible powder patterns are obtained after ignition of these minerals in certain gases under controlled temperatures. The current practice of heating a sample in a crucible over an open flame may lead to misinterpretation of resultant patterns.

Our studies have indicated that ignition of the noncrystalline specimens results in randomly oriented crystallized material. None of our samples recrystallized as single homogeneous crystals, although this phenomenon has been reported for gadolinite. The results also indicate that different gases have a marked influence on crystallization and on dissociation of some material.

The apparatus used for heating consists of a tube furnace that can be set up for heating in air or in any other atmosphere. The samples are first crushed and then heated at temperatures below their fusion points for a short time both in air and in uncontaminated helium. It was found that both helium and commercial nitrogen, which for a time was used as the inert gas, contain sufficient water to oxidize the specimen being heated.

The resulting *x*-ray powder patterns are strong with sharp diffraction lines indicating good crystallinity after ignition. Preliminary data on allanite, brannerite, davidite, fergusonite, samarskite, thorite and zircon are given.

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* Approved by Director of U. S. Geological Survey.

ROBINSONITE, A NEW LEAD ANTIMONY SULFIDE

L. G. BERRY, JOSEPH J. FAHEY, AND EDGAR H. BAILEY
Queen's University, Kingston, Ontario, U. S. Geological Survey,
Washington, D. C., and U. S. Geological Survey, Los Gatos, California

Triclinic; $a=16.51$, $b=17.62$, $c=3.97$A, $\alpha=96°04'$, $\beta=96°22'$, $\gamma=91°12'$. Slender prismatic [001], striated [001]; also massive, fibrous to compact. Cleavage, not observed; fracture irregular; brittle, H $2\frac{1}{2}-3$. Measured specific gravity, 5.27 (artificial crystals), 5.20 (natural fragments), 5.34 (artificial fused material). Composition and cell content, $7PbS \cdot 6Sb_2S_3$, with calculated specific gravity 5.40. Strongest x-ray powder lines in Angstroms: 4.08A(6), 3.97(6), 3.41(10), 3.19(6), 3.04(6), 2.74(5), 2.68(5).

Occurs as a primary mineral with pyrite, sphalerite, stibnite and boulangerite as small pieces in oxidized ore bodies at the Red Bird mercury mine, Pershing Co., Nevada.

PETROLOGY OF THE GUFFEY-MICANITE REGION, COLORADO

JAMES E. BEVER
University of Michigan, Ann Arbor, Michigan

The Guffey-Micanite region, which is in the southeast part of Park County and in the north-central part of Fremont County, Colorado, is underlain chiefly by pre-Cambrian rocks, bounded, except on the south, by extrusive igneous rocks of Tertiary age. The pre-Cambrian rocks include: (1) The Idaho Springs formation, which consists of mica schists and gneisses, sillimanite schists and gneisses, and lime-silicate rocks, all of meta-sedimentary origin. (2) Lenses and layers of hornblende gneiss and amphibolite that may represent metamorphosed gabbros or basalts. (3) Several bodies of granite, which may belong to more than one intrusive period. (4) Granitic pegmatites, related to the granite, containing the accessory assemblage: beryl (including golden beryl), garnet, apatite, columbite, schorl, Fe-Mn phosphates, magnetite, bismutite, beyerite, cordierite-pinite, sillimanite, monazite, and uranothorite (?). (5) Migmatite formed locally by intense injection of granitic material into rocks of the Idaho Springs formation. In addition to the pegmatite minerals, feldspar, sheet and scrap mica, beryl, and columbite, other minerals of economic significance include galena, sphalerite, scheelite, chalcopyrite (with anthophyllite), and sillimanite.

STRUCTURES IN ICE WEDGES OF NORTHERN ALASKA

ROBERT F. BLACK
U. S. Geological Survey, Washington, D. C.

Minute structures, generally considerably smaller and more complex than structures in glacial ice, were found in ground ice during petrofabric study of ground-ice wedges near Barrow, Alaska. Not all can be explained easily. However, results supplement concurrent research on ground contraction, temperatures, and composition and provide corroborative evidence for Leffingwell's contraction hypothesis of origin of ice wedges.

All wedges have myriad bands of air bubbles and inclusions of organic material and silt, sand, and gravel, which in most wedges produce marked foliation subparallel to the sides. Other bands crisscross within the wedges. Most air bubbles and inclusions are elongated vertically regardless of orientation of the band they are in.

All wedges show marked development of one or more microscopic growth fabrics in which c-axes are oriented about normal to cooling surfaces. The best-developed fabric, presumably resulting from recrystallization under pronounced temperature gradients and considerable lateral compression, is fan-shaped and conforms roughly to the shape of the wedge. Other fabrics are produced by growth of hoar and ice crystals in contraction cracks and tension fractures.

Most wedges also show one or more deformation fabrics superimposed on growth fabrics or vice versa. Individual grains respond to stress by strain, reorientation (apparently in part to permit slipping on the basal pinacoid or rolling around the *c*-axis), fracture, granulation, and recrystallization. Movement within wedges is indicated by granulated and recrystallized grains, tension fractures, shear planes, offset bands of air bubbles and inclusions, dragged sediments on sides of wedges, and surface ridges.

SYNTHETIC GRANITES AND THEIR MELTING BEHAVIOR UNDER HIGH H_2O PRESSURES

N. L. BOWEN AND O. F. TUTTLE
Geophysical Laboratory, Washington, D. C.

Mixtures of $NaAlSi_3O_8$, $KAlSi_3O_8$ and SiO_2 have now been examined as to their melting behavior at 4 isobars, 500, 1000, 2000 and 3000 kg/cm² pressure of water vapor. At each pressure it is found that the addition of SiO_2 to the feldspar solid solution series induces the expected further lowering of melting temperatures. The minimum melting temperature of the binary feldspar series is reflected in the ternary compositions as a minimum temperature on the boundary curve between the field of quartz and that of feldspar solid solutions. These minimum-melting mixtures are synthetic alkali granites.

The temperature of the minimum decreases with increasing pressure of water vapor, owing to the increased solubility of water in the liquid phase, but, whereas the first effects of increasing pressure are very marked, these diminish rapidly and the difference between the value for 2000 kg/cm² and that for 3000 kg/cm² is only about 5°, the actual temperatures being 675° and 670° respectively. Moreover, the composition of the minimum moves with increasing pressure towards a somewhat lower quartz content.

Some of the consequences of these observations on theories of the origin of granite will be discussed.

STUDIES OF RADIOACTIVE COMPOUNDS: IV—PITCHBLENDE FROM LAKE ATHABASKA

E. J. BROOKER AND E. W. NUFFIELD
Geological Survey of Canada, Ottawa; University of Toronto, Canada

Chemical analyses of six specimens of pitchblende from Lake Athabaska gave a range in U^6/total U from 17.4 to 85.0 per cent. With increasing U^6/U the specific gravity decreases from 8.20 to 4.10, the lustre becomes dull, and the mineral loses its hard, compact and brittle aggregation and assumes a soft, earthy form. Thus in a general way the physical properties are indicative of the degree of oxidation.

Specimens relatively low in U^6 gave sharp *x*-ray powder photographs. With increasing U^6/U, back reflections became weak and diffuse, then low angle reflections became diffuse and finally with $U^6/U = 85.0$ per cent no pattern was obtained. The corresponding cell edges decreased from 5.45 kX for $U^6/U = 17.4$ per cent as compared to 5.46 for synthetic UO_2, to 5.39 for $U^6/U = 78.5$ per cent. Therefore in the six specimens the quality of the *x*-ray pattern and the cell sizes are related to the degree of oxidation.

After heating for $\frac{1}{2}$ hour in vacuum the patterns were sharper and the cell edges decreased to 5.43 for synthetic UO_2, and to a minimum of 5.38 for the more highly oxidized specimens. In specimens with U^6/U about 50 per cent another oxide, possibly U_2O_5 also formed; this was replaced by U_3O_8 when U^6 dominates over U^4.

Heating for 5 minutes in air oxidized those specimens with U^6/U about 50 per cent to

U_2O_5, while those with a higher or lower U^6/U ratio were oxidized to U_3O_8. The cell edges of U_3O_8 increased with increasing availability of U^6 in the untreated material.

ISOTOPIC COMPOSITION OF LEAD AND THE AGES OF MINERALS IN A PRECAMBRIAN GRANITE

HARRISON BROWN, MARK C. INGHRAM, ESPER S. LARSEN, JR., CLAIRE PATTERSON AND GEORGE TILTON
University of Chicago, Chicago, Illinois; U. S. Geological Survey, Washington, D. C.

Techniques have been devised for the analysis of microquantities of lead and uranium in granitic materials using isotopic tracers. The general method consists of equilibrating known microamounts of lead or uranium highly enriched in a single isotope with known amounts of the sample, chemically isolating the element, and measuring the change in the isotopic composition of the tracer. Chemical procedures, which progressed from a macro- to a microscale, and surface ionization techniques in a mass spectrometer of a very high sensitivity were used. The concentration and isotopic composition of lead and uranium in mineral separates of a Canadian granite have been studied. These data have given the lead-uranium and lead-lead ages of the minerals. The data have also given the isotopic composition of a nonore lead of known Precambrian age, and the isotopic composition of nonore uranium.

ASSOCIATION OF PERTHITIC MICROCLINE WITH HIGHLY UNDULANT OR GRANULAR QUARTZ IN SOME CALCALKALINE GRANITES

FELIX CHAYES
Geophysical Laboratory, Washington, D. C.

Perthite is on the whole uncommon in the micaceous calcalkaline granites of New England, Texas, and the southeastern states. It is usually rare or lacking if quartz is ungranulated and shows only moderate undulance, may or may not be common if quartz extinction is highly undulant, and is never rare if quartz is extensively granulated. The shearing stresses which render quartz extinction undulant and finally granulate the quartz evidently induce or accelerate the unmixing of albite-poor microcline which might otherwise remain indefinitely metastable.

DISTINCTION BETWEEN THE DIFFERENT MEMBERS OF THE POTASH-FELDSPAR GROUP USING ONLY A MICROSCOPE

L. DOLAR-MANTUANI
University of British Columbia, Vancouver, British Columbia

As the different members of the potash-feldspar group have the same values of refractive indices and birefringence, overlapping values of the optic angles and finally an often only apparent triclinic orientation determined with a microscope, the following data should be used for their distinction:

1. the presence or absence of microscopically visible lamellar twinning;
2. the optic angle; 3. the extinction angles or coordinates of crystallographic elements; 4. the interfacial angles; 5. the habit; 6. the paragenesis indicating the conditions of crystallization.

The general properties and some limitations of their values as they may be determined with a microscope are listed for the various members. The terms perthite and potash-feldspar are briefly discussed. Detailed determination and publication of data especially of anomalous members are recommended.

AIDS FOR SINGLE-CRYSTAL TECHNIQUES

GABRIELLE DONNAY AND J. D. H. DONNAY
Geophysical Laboratory, Washington, D. C.; The Johns Hopkins University, Baltimore, Maryland

Two nomographs have been devised which permit rapid interpretation of rotation and Weissenberg photographs. One of them gives, from a rotation photograph, the direct lattice translation of the rotation axis. The other gives, from a zero-layer Weissenberg photograph, the reciprocal lattice translations. The use of the charts, when they are carefully constructed, entails no loss of accuracy.

Many crystals can have one of their lattice rows adjusted only approximately by optical means. A simple procedure has been worked out in which the corrections to be applied to the arcs of the goniometer head are obtained from a single film, on which a 10° oscillation with unfiltered radiation and a rotation with filtered radiation are superimposed. Measurement of the position of the continuous streak, due to the oscillation, with respect to the rotations record, leads to the desired corrections.

CRYOLITE TWINNING

J. D. H. DONNAY AND GABRIELLE DONNAY
The Johns Hopkins University, Baltimore, Maryland; Geophysical Laboratory, Washington, D. C.

The cubic pseudo-symmetry of a multiple cell leads to the prediction of many twin laws, most of which have been reported by Brögger. The submultiple cell used by Dorothy Wrinch has the same shape as the multiple cell and therefore leads to exactly the same twin laws, but it has a structural significance which the multiple cell does not possess. Her interpretation of twinning can be generalized in that the submultiple cell need not be pseudo-cubic, but can be pseudo-orthogonal. Its validity as a law of observation can only be established by further experimental evidence.

ORIGIN AND EVOLUTION OF HORNBLENDE-ANDESINE AMPHIBOLITES AND KINDRED FACIES*

A. E. J. ENGEL AND CELESTE G. ENGEL
California Institute of Technology, Pasadena, California

Nearly identical hornblendic amphibolites, in which andesine is commonly a major constituent, may evolve from: (1) skarnlike metasomatism of carbonate sediments and (2) reconstitution without appreciable chemical changes of tuffs and marls, gabbro masses, diorite sills, and basalt flows.

Commonly no obvious physico-chemical feature of these amphibolites offers an unequivocal clue to their origin or evolution. The amphibolites occur in most older pre-Cambrian terranes and in many younger orogens where each of the above-stated parent rocks and processes of formation are either apparent or readily inferred.

Clues to the derivation of these amphibolites seem to exist, however, in their differential inheritance of accessory elements. Amphibolites derived from mafic igneous rocks, for example, are, like their parents, commonly higher in Co, Ni, Cr, Sc, and Cu and lower in Pb, Au, and Ba than amphibolites derived from carbonate sediments.

Quantitative analyses of these elements in 82 parent rocks and amphibolites of known origin are discussed in conjunction with other features of the rocks. Apparently dynamothermal metamorphism of the gabbros does not remove the initial concentrations of Sc,

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* Publication authorized by the Director, U. S. Geological Survey.

Cr, Co, Ni, and Cu as rapidly as it obliterates other diagnostic properties. Conversely, amphibolites derived by replacement of marbles tend to maintain higher concentrations of Ba, Pb, and Au than many known ortho-amphibolites.

Moreover, the incipient granitization of amphibolites does not always obliterate the critical accessory-element differences at stages in which, by less subtle physio-chemical standards, various amphibolites are indistinguishable.

The accessory-element values in these amphibolites, and in some of their component minerals, are also compared with examples of enigmatic amphibolites from the Canadian shield, the Alps, and elsewhere.

NEW UNIVERSAL MICROSCOPE GONIOMETER

D. JEROME FISHER
University of Chicago, Chicago, Illinois

The instrument consists of a standard synchronous-nicol microscope supported on a triangular base plate so that its optical axis is horizontal with what was its "north-south" vibration direction inclined downward to the left (as viewed from the ocular) at 45°. The crystal is mounted on a standard x-ray camera goniometer head in the usual fashion. This head attaches to a vertical axis with the crystal extending downwards from it. The vertical axis, which is supported from the base plate independently of the microscope, is provided with two translation movements, one vertical, the other horizontal and normal to the microscope axis. The vertical axis has a graduated circle with vernier and tangent screw.

The crystal may be immersed in a suitable oil; this is held in a multiple cell of any convenient thickness. This cell which has thermocouple wires for temperature measurement fits in a frame supported on a vertical arm extending up from the base plate. The cell frame support is provided with two rack-and-pinion translations, one vertical, the other horizontal and normal to the microscope axis. The regular converger from the microscope substage is mounted "above" the stage so that it can be thrown in or out of the optical path; it is centerable and has a translation movement parallel to the axis of the microscope.

There is a detachable lamp housing with collimator and Websky slit, so that the instrument may function as a one-circle goniometer by using the Bertrand lens.

The instrument may be employed for optical orientation of an anhedron. Or conversely it may be used to locate the optical indicatrix of an anhedron whose orientation has been obtained by any x-ray method. It is superior to the Universal stage technique for these purposes, since it is more general; thus, no cleavage is needed for orientation purposes, and a single crystal with a single mounting is used for determining both the indicatrix and the lattice. Preliminary tests indicate it is superior to the U-stage in accuracy, also. The tremendous advantage gained by the use of synchronously rotating nicols should be obvious.

GEOLOGY OF THE CARRIZO MOUNTAIN SCHIST*

PETER T. FLAWN
The University of Texas, Austin, Texas

The name Carrizo Mountain schist has been applied to pre-Cambrian metasedimetary and meta-igneous rocks of the Van Horn area, Texas. The unit includes feldspathic metaquartzite and meta-arkose, mica schist, chlorite schist, phyllite, slate, limestone, metarhyolite (in part mylonitized), and amphibolite. Regional metamorphic grade increases from northwest to southeast. In the northwestern area a retrogressive metamorphism has followed the regional metamorphism.

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To the southeast the Carrizo Mountain schist is composed mainly of thoroughly recrystallized quartzofeldspathic rocks. Relict sedimentary structures are not evident. Associated almandine-bearing schists and amphibolites indicate a medium metamorphic grade. Crystalloblastic fabrics and equilibrium mineral assemblages characterize these rocks. To the northwest slate, phyllite, chlorite schist, and limestone occur with meta-quartzite and meta-arkose. Cross-bedding is preserved in the quartzofeldspathic rocks. Cataclastic fabrics, fine grain size, retrogressive disequilibrium relations, smearing, comminution, microfolding, and microfaulting are characteristic here. The retrogressive metamorphism occurred during the period of dislocation in which the Carrizo Mountain schist was thrust northwestward over the pre-Cambrian Allamoore limestone. Intrusive rhyolite diorite (now metarhyolite and amphibolite) were affected only by this later cataclastic metamorphism.

STUDY OF HOEGBOMITE

GERALD M. FRIEDMAN
University of Cincinnati, Cincinnati, Ohio

An investigation of the emery deposits of the Appalachian belt led to the discovery of a new hoegbomite locality in North Carolina, a reinvestigation of the Virginia hoegbomite and an examination of a New York locality from which hoegbomite has not been previously described.

Of the eight known hoegbomite occurrences seven have been reported in emery, the emery being as a rule associated with basic rocks. Hoegbomite is a secondary mineral that formed by the replacement of spinel.

The hoegbomite of the New York emery deposits was formed later than the emery but earlier than the high-temperature quartz veins which represent the last stages of the intrusion that gave rise to the emery.

ROLE OF THE HYDROGEN BOND IN THE FORMATION OF SOME METASTABLE PHASES

ALFRED J. FRUEH, JR.
University of Chicago, Chicago, Illinois

It has been well established that in the genesis of certain polymorphous mineral forms the pH of the crystallizing solution is a determining factor. However, the role of hydrogen has always been considered to be of a minor or secondary nature, such as causing the formation of new groups or complexes that add to or remove from the solution inhibiting impurities. Recent chemical research and a close inspection of structural relationships indicate that the formation of metastable phases in the case of some polymorphic pairs, such as senarmontite-valentinite, can be explained by the hydrogen bond. The closer oxygen-oxygen distances that exist in the high temperature form valentinite, can be maintained during the growth of this form, well below its stable temperature range, through the formation of O-H-O bonds. Because of the small energies involved in the formation and rupture of the hydrogen bond, it is not believed necessary to achieve or retain measurable proportions of hydrogen in the crystals produced by this mechanism. The involvement of chemically bonded hydrogen in one phase and its total absence in another restricts us from regarding the pair of phases in this instance as truly polymorphic; and consequently the generation of a phase containing hydrogen should not be considered a priori metastable.

MINERALOGY AND STRUCTURE OF THE LUZONITE-FAMATINITE SERIES

RICHARD V. GAINES
Harvard University, Cambridge, Massachusetts

New chemical analyses confirm that the formula of luzonite is Cu_3AsS_4 and that of famatinite is Cu_3SbS_4. They form an isomorphous series with the nearly pure end-members occurring, respectively, at the type localities for the two minerals. Intermediate members of the series are also found, at other localities. Crystals of luzonite from Mankayan, Luzon, Philippines, and of luzonite-famatinite from Goldfield, Nevada, were examined by goniometry and x-ray diffraction, and especially good x-ray results were obtained with fragments of famatinite from Sierra de Famatina, Argentina.

Luzonite-famatinite crystallizes in the tetragonal system, and luzonite is a polymorph of enargite. There are two cleavages, $\{100\}$ and $\{101\}$. Twinning is on $\{111\}$, and the twin axis is normal to $\{110\}$.

The space group is $I\bar{4}2m$, and the cell is pseudo-cubic, with $a_0=5.27$ Å and $c_0=10.39$ Å for luzonite, and $a_0=5.38$ Å and $c_0=10.76$ Å for famatinite. Powder photographs show a sphalerite type of structure. The structure has been tentatively worked out, and can best be compared for illustration with that of stannite, with which it is isostructural. To do this, arsenic atoms take the place of the iron atoms in stannite, and copper replaces the copper and tin atoms. This gives a body-centered tetragonal cell containing $Cu_6(AsSb)_2S_8$. Preliminary intensity calculations show this structure to be at least a very close approximation to the true structure.

SYENITES AND NEPHELINE SYENITES OF STETTIN, MARATHON COUNTY, WISCONSIN

ELAINE GEISSE
Smith College, Northampton, Massachusetts

A southern extension of the complex crystalline rocks of the pre-Cambrian Canadian Shield crops out in the Driftless Area of north-central Wisconsin, Stettin township, 8 miles west of Wausau, central Marathon County. A preliminary field survey was made of 6 square miles. A road-metal quarry in syenite (section 22, township of Stettin) offered excellent, fresh rock surfaces, in an area of otherwise scant and poor outcrops, where formerly only reconnaissance work had been done. Detailed study of the syenite quarry and adjacent $\frac{3}{4}$ square mile established (1) gray argillite, the original country rock; (2) "granite hybrid" with rounded included quartz grains; (3) nepheline syenite, bearing eucolite; (4) pink, fine-grained syenite apparently the main intrusive; (5) hornblende syenite in the pink syenite, possibly a darker phase of the syenite; (6) granite pegmatite containing zircon crystals. Nepheline was determined by treating slides with phosphoric acid and staining with methylene blue. A new occurrence of a mineral of the eudyalite-eucolite series was found and determination was made by thin section, fragment, and x-ray studies.

Nepheline syenites are often associated with limestones or carbonate rocks. There is no indication, up to the present time, of limestone in the Wausau area, although the argillite is rich in microscopic carbonate. These nepheline rocks and associated minerals resemble those of Magnet Cove, Arkansas; Haliburton-Bancroft, Ontario; Alnö, Sweden; and Oslo, Norway.

Further field and laboratory work is needed to determine whether these rocks are igneous, meta-igneous or meta-sedimentary in origin.

ORIGINAL CRUST IN THE CANADIAN SHIELD AREA

JAMES E. GILL
McGill University, Montreal, Canada

Archaean rock series in the Canadian Precambrian Shield include sediments, but none of the coarse clastics characteristically derived from granite. This is taken as direct evidence that the original crust was basic—probably basaltic, and that granite was scarce, if not entirely absent. The granitic rocks so abundant in the Shield developed and became exposed through repeated cycles of sedimentation, mountain building, and erosion.

NEW BASIC COPPER PHOSPHATE MINERAL FROM SANTA RITA, NEW MEXICO

CARL W. BECK AND DAVID B. GIVENS
University of New Mexico, Albuquerque, New Mexico

Crystallographic studies of small, emerald-green crystals from the Santa Rita pit, Chino Mines Division, Kennecott Copper Corporation, Santa Rita, New Mexico, lead to the description of a new mineral dimorphous with pseudomalachite, $Cu_5(PO_4)_2(OH)_4$.

X-ray studies by the Weissenberg method on the new mineral showed it to be orthorhombic and gave a space group $Pnn—C_{2v}^{10}$ with cell dimensions $a_0 = 7.47$ Å, $b_0 = 8.31$ Å, $c_0 = 5.83$ Å and axial ratios $a:b:c = 0.8989:1:0.7016$. The unit cell contents are $Cu_{10}(PO_4)_4(OH)_8 = 2[Cu_5(PO_4)_2(OH)_4]$.

The optical data are: $\alpha = 1.698$, $\beta = 1.745$, $\gamma = 1.783$; $2V = 90° \pm$; optically $(+)$; $X = c$.

Crystal forms present are $b\{010\}$, $m\{110\}$, $n\{011\}$. Cleavage is $\{110\}$, perfect.

The calculated specific gravity is 5.24 and the measured specific gravity is 5.22.

AUROSTIBITE–A NEW MINERAL IN THE PYRITE GROUP*

A. R. GRAHAM AND S. KAIMAN
Department of Mines and Technical Surveys, Ottawa, Canada

A new mineral identical with the intermetallic compound $AuSb_2$ has been found in gold ores from the Giant Yellowknife Mine, North-West Territories, and from the Chesterville Mine, Larder Lake Area, Ontario. In the former locality it occurs in dolomitic carbonate and quartz with gold, freibergite, stibnite, jamesonite, chalcostibite, bournonite, arsenopyrite, pyrite, chalcopyrite, and sphalerite; in the latter in quartz with gold, galena, tennantite, chalcopyrite, sphalerite, arsenopyrite, gersdorffite and pyrite. In polished section the mineral is galena-like with a slight pinkish tinge; in hand specimen minute anhedral grains show a bornite-like tarnish. The standard etch reactions in the order of reactivity are: HNO_3, $FeCl_3$, HCl, KOH positive; $HgCl_2$ and KCN negative. The hardness is C−. The composition of the mineral was determined by comparing its *x*-ray powder pattern with that of the synthetic compound, and was checked by microchemical tests. The crystal structure is pyrite type; the unit cell containing Au_4Sb_8 has $a = 6.646 \pm 0.003$ kX. (synthetic 6.644 ± 0.003 kX); the single Sb parameter is 0.386 ± 0.007. The calculated gravity is 9.91 compared with 9.98 measured on synthetic material.

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PETROLOGY OF THE PALEOZOIC SHALES OF ILLINOIS*

R. E. GRIM, W. F. BRADLEY, W. A. WHITE
State Geological Survey, Urbana, Illinois

A series of Paleozoic shales ranging from the Ordovician through the Pennsylvanian from widely separated geographic localities in Illinois were investigated by *x*-ray diffraction, differential thermal, chemical, and microscopic methods, in order to determine their clay mineral composition and textural characteristics.

Illite is the dominant clay mineral in most of the shales investigated, a chloritic clay mineral is present in many of them in varying abundance, kaolinite is a component of many of them in varying abundance, and montmorillonite was found in only a few of them in minor quantities.

An analysis of the data shows that certain clay mineral compositions are characteristic of certain parts of the Paleozoic section in Illinois. A discussion is presented of the relation of clay mineral composition to the texture of shales and of the implication of the relation of composition to the genesis of the shales.

VARIATIONS IN GRANITIC ROCKS OF THE HUNTINGTON LAKE AREA OF THE SIERRA NEVADA

WARREN B. HAMILTON
University of Oklahoma, Norman, Oklahoma

The rocks of the Huntington Lake area, 160 square miles of the western slope of the central Sierra Nevada, of California, are dominantly granitic. Two square miles of the area are underlain by metamorphic rocks, chiefly quartzites and marbles.

The granitic rocks vary from calcic diorite to alkali alaskite and form twelve separate plutons. Each of these plutons varies within itself, and a series of maps has been prepared illustrating the following variables: rock type, abundance and relative proportions of ferromagnesian minerals, abundance of dark inclusions, grain size, and quality of primary flow structures. Gradational variations within plutons are of considerable magnitude in some cases; in two plutons, for example, rock type varies from quartz diorite through granodiorite to quartz monzonite. In another pluton, content of ferromagnesian minerals varies from two to nineteen per cent. The "typical" granitic rock would be a hornblende biotite quartz monzonite.

Total dark minerals, soda lime feldspar, and the proportion of hornblende to biotite in general increase together; precise statements regarding their interrelation are not warranted.

The abundances of ferromagnesian minerals and of dark inclusions are closely parallel. The inclusions probably have a xenolithic origin.

PETROLOGY OF THE FRANKLIN-SYLVA PEGMATITE DISTRICT, NORTH CAROLINA

E. WM. HEINRICH
University of Michigan, Ann Arbor, Michigan

The Franklin-Sylva pegmatite district, which includes parts of Clay, Macon, Jackson, Transylvania and Haywood counties in North Carolina, is underlain in its western two-thirds chiefly by high-grade metamorphic rocks and in its eastern one-third mainly by the Whiteside batholith, which varies in composition from quartz dioritic to granodioritic and by whose crystallization the mica-bearing pegmatites were derived. The metamorphic rocks, which were studied by Keith, were divided by him into two formations: the Carolina gneiss, which included mainly biotitic gneisses and schists, and the Roan gneiss, which he described as dominantly hornblende gneiss and schist. More detailed work has revealed a much wider variation in rock types. Rocks previously classed together under Carolina gneiss include: biotite gneiss, biotite-garnet gneiss, biotite-muscovite gneiss ± garnet, quartz-biotite gneiss ± garnet, feldspathic biotite gneiss, garnetiferous quartzite, kyanite gneiss ± garnet or staurolite, kyanite-sillimanite gneiss and schist, sillimanite-biotite gneiss ± garnet, a series of rocks containing variable amounts of garnet (rhodolite), anthophyllite, biotite, hypersthene, sillimanite and quartz, a group of rocks containing corundum in various combinations with garnet, biotite, kyanite, hypersthene, hornblende or chlorite, and locally, minor marble. The Roan gneiss is now known to contain hornblende gneiss and schist ± garnet, amphibolite, garnet amphibolite and actinolite schist. This complex of probable pre-Cambrian age, was intruded first by bodies of dunite, troctolite and peridotite, which display various degrees of serpentinization and complex hydrothermal alteration. Later, possibly in Carboniferous time, the Whiteside batholith with its satellitic pegmatites and aplites was emplaced, and migmatites were developed locally in the schists and gneisses through intense, small-scale injection.

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NOTES ON THE GEOCHEMISTRY OF BERYLLIUM*

W. T. HOLSER, L. A. WARNER, V. R. WILMARTH, AND E. N. CAMERON
Cornell University, Ithaca, New York; University of Colorado, Boulder, Colorado; U. S. Geological Survey, Denver, Colorado; University of Wisconsin, Madison, Wisconsin

Extensive sampling of nonpegmatitic rocks by the U. S. Geological Survey has provided many new data that suggest modification of prevailing concepts of beryllium geochemistry.

Common silicates, particularly those characterized by tetrahedrally coordinated aluminum, contain as much as 0.0X per cent BeO. Substitution of $Be^{2+}Si^{4+}$ for $Al^{3+}Al^{3+}$ is probable. Idocrase is exceptional in that it can contain a maximum of nearly 4 per cent BeO. When shown on a triangular diagram with coordination-number coordinates, nonberyllian idocrase analyses are grouped near Machatschki's formula $(Ca_{19}(Al, Mg)_{13}Si_{18}(O, OH, F)_{76})$. Idocrase with more than 0.1 per cent BeO lies far outside the normal field, but if beryllium is not included in the calculation the plots are normal. Probably a third of the beryllium replaces silicon; the rest enters normally unoccupied tetrahedral positions. Beryllian idocrase is known only at localities where beryllium minerals such as helvite are found. Garnet is similar to idocrase in structure, but it contains less than a tenth as much beryllium.

Igneous rocks vary widely in beryllium content. New tentative averages are: 0.0025 per cent BeO in feldspathoidal rocks, 0.002 in granitic rocks, and 0.0007 in intermediate rocks. Feldspathoidal intrusive rocks in the United States, including the rare pegmatitic phases, do not contain beryllium minerals. Beryllium is apparently accommodated in the lattices of aegerite and nephelite.

About 15 per cent of the pyrometasomatic deposits in the United States contain tactites with more than 0.005 per cent BeO, but none approach in size and grade the Iron Mountain, N. Mex., deposit. The association of idocrase, usually fluorine-rich, with fluorite is indicative of beryllium.

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* To appear as part of a Geological Survey bulletin "Non-pegmatite beryllium resources of the United States."

ON THE SUPERSTRUCTURE IN ANORTHITE

FRITZ LAVES AND JULIAN R. GOLDSMITH
University of Chicago, Chicago, Illinois

Taylor and his coworkers (1934, 1951) have shown that in anorthite the true periodicity in the *c*-direction is approximately twice that of albite. Additional observations here reported may be helpful in the final determination of the anorthite structure, considerable differences are observed with variation of heat treatment. Anorthites from several localities as well as synthetic anorthite show three types of reflections:

(a) (hkl) with l = even and (h+k) = even
(b) (hkl) with l = odd and (h+k) = odd
(c) (hkl) with (h+k+l) = odd
    (1) (h+k) = even, l = odd
    (2) (h+k) = odd, l = even

Type (a) spots correspond to those of the albite structure, are always sharp, and unaffected by heat treatment. Types (b) and (c) are affected by history and heat treatment. (b) and (c) can be diffuse, although (c) reflections are elongated in the direction normal to (010), when diffuse.

If all of the spots are or have been made sharp (6 days at 1150° C. for synthetic anorthite) the average ratio of the intensities of (a)/(c, 1)/(b), (c, 2) is approximately equal to 20/5/1. In 15 minutes at 1500° C. the (c) spots virtually disappear so that the average intensity (a)/(c)/(b) is 20/0/1. (The apparent disappearance of the (c) spots may be due to greatly increased diffusion, and must be checked photometrically.)

It is believed that these observations are best explained by assuming the (a) spots to be due to a fundamental plagioclase structure. The subsidiary spots (b) and (c) are a consequence of order-disorder relations with Al-Si ions controlling the (b) reflections, Ca ions the (c) reflections.

Additional data obtained from synthetic anorthites in which partial replacements were made of Al by Ga, of Si by Ge, and of both Al and Si by Ga and Ge confirm this hypothesis.

PROBLEM OF THE CARBONATE APATITES. IV—STRUCTURAL SUBSTITUTIONS INVOLVING CO_3 AND OH

DUNCAN McCONNELL
The Ohio State University, Columbus, Ohio

That the formula for francolite cannot be written correctly as $3Ca_3(PO_4)_2 \cdot CaCO_3$ is conclusively demonstrated by the fact that the atomic ratios Ca:P:C are not constant and do not closely approximate 10:6:1. Although certain authors have presumed that francolite consists of two phases, namely fluorapatite and $CaCO_3$, this presumption is inconsistent with most of the experimental data.

Optical and *x*-ray diffraction data completely fail to indicate the presence of a second $CaCO_3$ phase. If it is assumed that the $CaCO_3$ particles are so small as to produce no diffraction lines, one would expect the francolite lines to be broad and diffuse because the dimensions of the $CaCO_3$ particles could not be less than one-tenth the dimensions of the francolite particles. The diffraction lines of francolite are quite as sharp and narrow as those of apatite, however.

Although the diffraction patterns of apatite and francolite are similar, important differences exist. The optical properties are different also. Thus the basic structure of francolite must be similar to that of apatite, but certain statistical substitutions are necessary. Crystal chemical calculations indicate the following isomorphic substitutions: (i) C for P, i.e. $3PO_4 \rightarrow 4CO_3$, (ii) $(OH)_4$ for PO_4, and probably (iii) H_2O for Ca. This hypothesis is consistent with the average composition for five recent analyses of francolite, a fact which can hardly be fortuitous.

These concepts have important implications concerning the crystal chemistry of teeth and bones, because of the necessity for explaining the carbonate content of the substances in view of their apatite-like diffraction patterns.

HURLBUTITE, $CaBe_2(PO_4)_2$, A NEW MINERAL

MARY E. MROSE
State Teachers College, Salem and Harvard University,
Cambridge, Massachusetts

A new calcium beryllium phosphate closely associated with triphylite has been found on the dump at the Smith Mine, Chandler's Mill, Newport, New Hampshire. The original find of three crystals (summer of 1949) has recently been increased to twenty-two.

Hurlbutite is orthorhombic-dipyramidal; crystals are colorless to greenish white, 4 mm. to 25 mm. along [110], stout prismatic [001] with observed forms {001}, {010}, {110}, {201}, and {311} doubtful. The faces often are delicately etched and show striations on {110}. Cleavage, not observed. Fracture conchoidal. Brittle. Hardness 6. Specific gravity 2.877 (meas.); 2.88 (calc.). Luster vitreous to greasy. Transparent to translucent. Optically biaxial negative (−) with $nX = 1.595$, $nY = 1.601$, $nZ = 1.604$; $2V = 70°$; $r > v$, weak; $X = b$ $Y = c$. Chemical analysis gave: CaO 21.84, BeO 21.30, P_2O_5 56.19, Insol. 0.76, Total 100.09.

X-ray study by the Weissenberg method about all three axes was made on a single fragment of known orientation, drilled out from the original crystal found, following the location of the axes by the oscillating technique. Space group *Pmmm*. Unit cell dimensions a_0 8.29 Å, b_0 8.80, c_0 7.81 ($a_0{:}b_0{:}c_0 = 0.9420{:}1{:}0.8875$). The strongest *x*-ray powder lines are 3.67 Å (10), 3.03 (9), 2.78 (9), 2.21 (9).

Hurlbutite occurs in a pegmatite of the complex acid type with lithium, beryllium, and phosphate phases. The mineral assemblage includes muscovite, albite, triphylite, and massive light-smoky quartz, with indications that hurlbutite is later than triphylite but earlier than quartz. Perfect crystals are usually embedded in the smoky quartz. Secondary minerals include beryllonite, albite, brazilianite, twinned amblygonite crystals, colorless and purple apatite, siderite, and siderite pseudomorphs after hurlbutite.

THE UNIT CELL OF HYDROMAGNESITE

JOSEPH MURDOCH
University of California at Los Angeles

X-ray study of crystals of hydromagnesite from Crestmore, California, by powder, rotation and Weissenberg equi-inclination photographs, shows the following values for the crystallographic constants:

Monoclinic, pseudo-orthorhombic

$$a_0 = 18.69\text{Å} \quad b_0 = 9.02\text{Å} \quad c_0 = 8.33\text{Å}$$
$$a{:}b{:}c = 2.0720{:}1{:}0.9235 \; \beta = 90°$$

These values represent a doubling of the original *a* axis. Rotation and Weissenberg pictures were taken about [001] and [010]. The layer line (equator, first and second layers) photographs show that the original *a* axis must be doubled, and also show systematic extinctions which apparently place the symmetry in class C_{2v}^{14} (orthorhombic). This apparent symmetry is produced by polysynthetic twinning on {100} which fails to appear in the layer line pictures because of the orthorhombic aspect of the elementary lattice.

The powder photographs can be satisfactorily indexed using the new cell dimensions.

Values for $\frac{1}{2}a_0:b_0:c_0$, and spacings and intensities of the powder photographs correspond closely with those determined by Fenoglio in 1936 on hydromagnesite from Pennsylvania and from Val d'Aosta.

OBSERVATIONS ON THE STRUCTURE OF SOME KAOLINITES AND DEHYDRATED HALLOYSITE

HAYDN H. MURRAY
Indiana University, Bloomington, Indiana

The structures of several kaolinites and dehydrated halloysite, as indicated by x-ray powder diffraction technique and differential thermal analyses, have some significant variations. These variations are found within the unit structural layer and also in the manner in which the layers are stacked one upon the other. The term "degree of crystallinity" is used to compare the structural variations and is defined to include the disorder within the crystallographic unit layer and also the stacking variations of these unit layers.

The kaolinites and dehydrated halloysite were listed according to their degree of crystallinity first using criteria based on x-ray powder diffraction data and then using criteria based on differential thermal analyses. These two lists were compared and are in general agreement with the major exception of dehydrated halloysite.

Based on x-ray criteria, dehydrated halloysite has a poor "degree of crystallinity"; but based on differential thermal criteria, it has a good "degree of crystallinity." This indicates that the unit layer has very little disorder but that these layers are stacked with complete randomness.

AGE OF MINERALIZATION: THERMOLUMINESCENCE AND RADIOACTIVITY OF FLUORITE

JAMES M. PARKS, JR., AND DONALD F. SAUNDERS
University of Wisconsin, Madison, Wisconsin

The intensity of thermoluminescence of fluorite per unit radioactivity appears to be a function of the age of mineralization of the fluorite deposit. The intensity of thermoluminescence is proportional to the total radiation (alpha particle bombardment) received by the crystal lattice from radioactive impurities in the fluorite. The total radiation received by a given sample is a function of the present radioactivity of the specimen and its age, or the length of time since its formation.

About twenty fresh (not exposed to sunlight) samples of fluorite were obtained from the principal fluorite-producing areas in the United States, and from other mineralized areas where fluorite was available and the approximate geologic age of the mineralization was known. The procedures and apparatus for measuring the thermoluminescence and the radioactivity of the fluorite samples will be discussed briefly.

The fluorite samples used in this study range in geologic age from Precambrian to Tertiary. In most cases, the geologic evidence can only indicate an age as "post-this" and "pre-that," with perhaps a gap of a geologic period or more between the limits. With this new method involving the thermoluminescence and radioactivity of fluorite, the time of mineralization can generally be placed within a geologic period or a part thereof, according to the generally accepted absolute time scale of the National Research Council.

The possible sources of error, corrections involved, and the accuracy of this method will be discussed briefly. This general method is probably applicable to all thermoluminescent minerals, including calcite, dolomite, feldspar and quartz.

MAGNETIC SUSCEPTIBILITY OF CHROMITE FROM MONTANA AND ITS RELATION TO OTHER PHYSICAL AND CHEMICAL PROPERTIES

JOE WEBB PEOPLES AND GORDON P. EATON
Wesleyan University, Middletown, Connecticut

Because qualitative tests have indicated considerable variation in the magnetic properties of chromite from the Stillwater Complex, Montana, a study was made of the magnetic susceptibility of cleaned chromite concentrates previously analyzed. The magnetic susceptibility, at a field strength of 6400 gauss, ranges from 5.16×10^6 to 121.94×10^6 Cgs units for 33 chromite samples from the Stillwater Complex, but for one sample from Red Lodge, Montana, it is 505×10^6 Cgs units. Complete analyses of 10 of the samples, and determination of Fe and Cr for the rest had previously been made by R. E. Stevens and M. K. Carron of the U. S. Geological Survey. Preliminary microscopic study shows little evidence of nonhomogeneity; the magnetic properties are, therefore, considered as a property of the chromite itself. In general no correlation between magnetic susceptibility and weight per cent of total Fe is evident. Eight of the ten samples analyzed for all major constituents show a linear relationship between magnetic susceptibility and the ratio Fe(mol)/Fe(mol) +Mg(mol).

The variations of the physical properties and geologic occurrence of the magnetic and nonmagnetic chromite samples are compared with those of chromite from other deposits.

NEW MECHANICAL TWINNING IN CALCITE

EUGENE C. ROBERTSON
Harvard University, Cambridge, Massachusetts

Cylinders of calcite were caused to deform by subjecting them to hydrostatic pressure plus additional compression parallel to the axes of the cylinders. The test specimens were right circular cylinders, one-half inch in diameter, cut from single crystals of calcite; in one orientation the cylinder axes were perpendicular to a cleavage face, $(10\bar{1}1)$, and in another orientation the axes were parallel to a crystallographic *a*-axis. The resulting deformations were by twin gliding on the prism: $T=\{10\bar{1}0\}$, with two directions of gliding on the twin plane, $t_1=(0001)$, and $t_2=(000\bar{1})$. Slipping on the twin plane for relatively large distances was observed, and may be a parting mechanism of deformation with self-healing on the slip plane. The specimens were jacketed to prevent weakening by penetration of the pressure fluid into cracks. The shearing stress on the twin plane at the initiation of copious strain was not constant because of the irregularities of stress distribution in the cylinders, especially near the ends. A low hydrostatic pressure, less than 250 atmospheres, seems to be required before the twinning can be produced.

COMPOSITION AND SIGNIFICANCE OF FLUID INCLUSIONS IN MINERALS

EDWIN ROEDDER
University of Utah, Salt Lake City, Utah

During the formation of many minerals small amounts of the fluid medium from which deposition occurred are trapped within the growing crystals as microscopic fluid inclusions. These inclusions thus represent a true sample of the original medium from which deposition has taken place, and any information, however meager, which can be obtained from them as to the chemical nature of these fluids would be of considerable value in such diverse problems as the origin of cherts, the identification and correlation of veins or epochs of deposition in faulted or complex mining areas, the origin of certain detrital sediments, and the origin and nature of the fluids which have deposited pegmatites, quartz veins, and the ores of the metals.

A technique has been developed for the extraction and limited wet chemical and spectrographic analysis of the fluid from fluid-filled inclusions in quartz. At present it appears adequate to yield significant data on the sulfate:chloride ratio, the K:Na ratio, and certain spectrographic determinations. The technique and its limitations are discussed and results are given for quartz samples from a number of widely divergent types of occurrence, along with their significance in connection with the nature of the ore-forming fluid.

STUDIES OF MINERALS IN DUNITES AND IN OLIVINE-RICH INCLUSIONS IN BASALTIC ROCKS

C. S. ROSS AND MARGARET D. FOSTER
U. S. Geological Survey, Washington, D. C.

Dunites and olivine-rich inclusions in basaltic rocks, both of world-wide provenance and both characterized by the same four minerals—olivine, enstatite, diopside, and a spinel—provide an unusual opportunity to compare the composition of the minerals, and the distribution of the elements in them. These relationships contribute to an understanding of the genetic history of such rocks.

Materials were obtained from 14 widely distributed localities, and the various minerals were carefully separated for detailed mineralogical and chemical study. The analyses showed that the respective silicate minerals in olivine-rich inclusions and in dunites were strikingly similar in composition. This was particularly true of the olivine, in which, for all the specimens studied, there was a range of only 1 per cent in the silica content and of only 2 per cent in the magnesia. Much greater differences were found in the composition of the spinel, particularly in that in olivine-rich inclusions, which differed greatly in iron, chromium, and, especially, in alumina content. The samples of spinel from dunites were very similar to each other and were much higher in chromium than were those from olivine-rich inclusions.

Their striking mineral identity suggests that the original source both of dunites and of olivine-rich inclusions is the same and that the inclusions are derived from the peridotite zone of the earth's crust, as has been commonly believed for dunites.

DETERMINATION OF SMALL QUANTITIES OF DOLOMITE BY DIFFERENTIAL THERMAL ANALYSIS

RICHARDS A. ROWLAND AND CARL W. BECK
Shell Oil Company, Houston, Texas

In a furnace atmosphere of CO_2 the two endothermic loops of dolomite are separated and sharply defined. The first, between 700° C. and 800° C., is formed during the decarbonation of the magnesium carbonate. The second, between 900° C. and 1000° C., accompanies the decarbonation of calcium carbonate and coincides with the thermogram loop for calcite. The ratio of the areas enclosed by these loops and the extended base line is directly proportional to the dolomite content of a carbonate rock, and the area of the first loop reflects the dolomite content of other rocks when only a small amount of dolomite is present. Calibration curves are obtained by plotting these areas or area ratios from thermograms of prepared mixtures against the known dolomite content. For mixtures containing less than five per cent dolomite, the first loop is recorded at a sensitivity about thirty times as great as the sensitivity used for the second loop. The change from high to low sensitivity is made at 850° C. This method, by which the dolomite content of a carbonate rock can be quantitatively determined when as little as 0.3 per cent dolomite is present, is most useful in following dolomitization far beyond the limits of microscopy or *x*-ray diffraction and in evaluating the products of experiments in the laboratory synthesis of dolomite.

THE SYSTEM MgO-Al_2O_3-H_2O

D. M. ROY AND E. F. OSBORN
Pennsylvania State College, State College, Pennsylvania

Phase equilibria in the binary systems MgO-H_2O and MgO-Al_2O_3 were investigated. New data in the former system indicate that the transformation $Mg(OH)_2 \leftrightarrows MgO + H_2O$ takes place from 700° to 800° C. varying within the range of water vapor pressures studied (2000–20,000 p.s.i.). In the system MgO-Al_2O_3 spinel solid solutions were formed containing up to 85 mole per cent Al_2O_3. Exsolution of the solid solutions was accomplished under both dry and hydrothermal conditions. The extent of solid solution was found to decrease with decreasing temperature, the limit of solid solution being 65 mole per cent Al_2O_3 at 1500° C., and exsolution being almost complete at 800° C. in the presence of water vapor.

Under hydrothermal conditions, spinel was formed at temperatures between 900 and 375° C. At temperatures lower than 375° C. it is replaced by brucite+diaspore. Hydrotalcite ($6MgO \cdot Al_2O_3 \cdot CO_2 \cdot 12H_2O$), a mineral occurring naturally as a decomposition product of spinel was synthesized below 310° C. under varying H_2O and CO_2 pressures. A nitrate analogue of hydrotalcite was also synthesized at temperatures below 420° C. under hydrothermal conditions. This compound was found to coexist with spinel between 420° and 375° C. below which the spinel no longer forms.

PHASE EQUILIBRIA IN THE SYSTEM Al_2O_3-SiO_2-H_2O

RUSTUM ROY AND E. F. OSBORN
Pennsylvania State College, State College, Pennsylvania

The equilibrium assemblages of phases in the system alumina-silica-water have been studied under hydrothermal conditions over the range of 100° to 950° C. and 15 to 30,000 psi. Seven curves of univariant equilibrium are presented on a pressure-temperature diagram, representing the conditions at which one set of stable phase assemblages gives way to another. Two new phases are reported, one a pure aluminian end member of the montmorillonites and the other a mica-like phase formed on the decomposition of kaolinite.

The data serve to establish the stability relationship of endellite ($Al_2O_3 \cdot 2SiO_2 \cdot 4H_2O$) to halloysite ($Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$). The decomposition temperature of endellite changes from just above 100° C. at 15 psi. water pressure to nearly 200° C. at approximately 10,000 psi. The equilibrium decomposition temperature of the kaolinite group of minerals was found to be 450° C. and is very similar for the various members of the group. Pyrophyllite is a stable phase in equilibrium with a fluid in the system $Al_2O_3-SiO_2-H_2O$ from 420° to 575° C. At temperatures above 575° C. mullite appears as a phase in all mixtures. It was not found possible to synthesize andalusite, sillimanite, or kyanite under any of the conditions used; however, these minerals were successfully decomposed to yield equilibrium phases. This latter decomposition was achieved in an apparatus for grinding at elevated temperatures (up to 500° C.) and pressures (up to 10,000 psi.).

SYSTEM FeO-Al_2O_3-SiO_2

J. F. SCHAIRER AND KENZO YAGI
Geophysical Laboratory, Washington, D. C.

Phase equilibrium studies of this system, at and just below temperatures where a liquid phase is present, show one ternary compound, the iron analogue of cordierite ($2FeO \cdot 2Al_2O_3$

$\cdot 5SiO_2$), which decomposes at 1210±10° C. to mullite, tridymite, and liquid. The fields of stability of corundum, mullite, hercynite, iron cordierite, cristobalite, tridymite, fayalite, and wüstite have been delineated. The following invariant points (the first two eutectics and the remainder reaction points) were located:

Fayalite+wüstite+hercynite+liquid	1148± 5°
Fayalite+iron cordierite+tridymite+liquid	1083± 5°
Fayalite+iron cordierite+hercynite+liquid	1088± 5°
Hercynite+iron cordierite+mullite+liquid	1205±10°
Iron cordierite+mullite+tridymite+liquid	1210±10°
Corundum+mullite+hercynite+liquid	1380± 5°
Cristobalite+tridymite+mullite+liquid	1470±10°

Iron cordierite crystallizes with some reluctance and the metastable invariant points fayalite+tridymite+spinel+liquid at 1073±5° and mullite+hercynite+tridymite+liquid at 1205±10° can be realized. No ferrosilite or almandine garnet could be crystallized from the melts at any temperature even when melts were seeded with these crystalline phases. Natural almandine from Botallack, England (91.3 per cent almandine), when heated begins to decompose at an appreciable rate as low as 900° C. and yields a mixture of hercynite, iron cordierite, and fayalite. The bearing of these results on petrology and slags is discussed.

MARGARITE-EPHESITE SERIES; THEORY VERSUS FACT*

W. T. SCHALLER AND M. K. CARRON
U. S. Geological Survey, Washington, D. C.

The formula of margarite, the best known of the brittle micas, $Ca \cdot Al_2 \cdot Al_2Si_2 \cdot O_{10}(OH)_2$, requires 14.09 per cent CaO. Yet not a single one of nearly 5 dozen analyses shows this much CaO.

The presence of several per cent of Na_2O in many margarites has long been known—the soda margarites. Substituting one sodium for one calcium in the margarite formula leaves a deficiency of one in the cation charges. Theory calls for the addition of one atom of a univalent element whose ionic radius is close to that of aluminum in order to balance the charges. Lithium is the only element which meets these requirements and it goes into the only vacant position, in the octahedral group, of the margarite formula. The theoretical formula of the calcium-free sodium analogue of the margarite formula thus becomes $Na \cdot Al_2Li \cdot Al_2Si_2 \cdot O_{10}(OH)_2$, the mineral ephesite.

Now what are the facts? The calculated amount of Li_2O in the ephesite formula is 3.85 per cent. The two recorded analyses of ephesite from Postmasburg, South Africa, show only 0.9 and 1.5 per cent Li_2O. Either the theory is wrong or the reported analytical determinations of Li_2O are wrong. A new analysis of this ephesite was therefore made and 3.80 per cent Li_2O was found, thus confirming the theoretical approach to the correct formula of ephesite.

CHLORITE-CALCITE PSEUDOMORPHS AFTER ORTHOCLASE PHENOCRYSTS, RAY, ARIZONA

GEORGE M, SCHWARTZ
University of Minnesota, Minneapolis, Minnesota

Peculiar dark pseudomorphs after orthoclase phenocrysts occur along fracture zones near the margin of the metallized area at Ray, Arizona. The most intensely altered crystals contain no recognizable orthoclase but consist principally of chlorite and calcite plus small, but variable amounts of sericite and quartz. Rutile, leucoxene, kaolinite, allophane, hydromica and sulfides are present in some thin sections.

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* Publication authorized by the Director, U. S. Geological Survey.

It is thought that the pseudomorphs were formed by solutions loaded with calcium, magnesium and iron that moved upward and outward from the orebody where the rocks had been intensely sericitized and the bases other than potassium removed to a considerable degree.

The occurrence emphasizes the problem of the time of alteration. The propylitic stage is often considered to be an early stage, whereas it probably is often a marginal stage that coincides in time with sericitization, or other alteration, near the center of activity. An expanding sericite-quartz stage of alteration may cause those minerals to invade the propylitic rock and thus furnish evidence of an age difference, but in fact propylitization is probably taking place farther out at the same time.

CORONITES FROM THE PRESTON GABBRO, NEW LONDON COUNTY, CONNECTICUT*

CHARLES B. SCLAR
Battelle Memorial Institute, Columbus, Ohio

Corona-bearing rocks from the olivine hyperite facies and the troctolite facies of the Preston gabbro are described. Double coronas are present between olivine and plagioclase and between iron ore and plagioclase; single coronas are found between late-magmatic iron ore and plagioclase. The inner shell of the double corona consists of a colorless fibrous mineral (orthopyroxene, anthophyllite, or cummingtonite) that was derived at least in part from magmatic reaction rims of hypersthene that surround the olivine. The outer shell is composed of fibrous actinolitic amphibole that developed partly at the expense of deuteric(?) brown hornblende but mainly by replacement of the adjoining plagioclase. Enigmatic double coronas between plagioclase and iron ore were originally formed between plagioclase and olivine that was later selectively replaced by iron ore. Partly altered magmatic reaction rims of hypersthene surrounded by fully developed outer shells of actinolitic amphibole suggest that the inner shells of many double coronas were formed after the peripheral shells. The single coronas are identical with the outer shells of the double coronas and have the same developmental history.

The coronites grade into more highly altered equivalents in which the outer shells of the double coronas remain intact and the inner shells are replaced by either talc or chlorite and actinolite.

Field evidence does not support Shand's hypothesis of thermal metamorphism as the catalytic factor in corona formation. These coronites are believed to have developed as a result of hydrothermal alteration within a critical temperature range of chemically and mineralogically favorable rocks.

It is proposed that the term "corona" be limited to shells of post-magmatic synantectic minerals. Criteria for distinguishing magmatic from post-magmatic reaction rims based on mineralogical composition and fabric relationships are discussed.

RELATION OF MICROPEGMATITE TO PEGMATITE AT SAFE HARBOR, PENNSYLVANIA

W. HAROLD TOMLINSON
Springfield, Pennsylvania

At Safe Harbor, Pa., Antietam sediments containing pegmatite pockets and veins have been intruded by basaltic magma. The sediments vary in composition from compact mica

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* Published by permission of the Director, Connecticut Geological and Natural History Survey.

schist strata to more porous arenaceous strata composed of quartz and felsitic minerals. Part of the aureole has been entered by sodic vapors which penetrated readily through the arenaceous strata, less freely through the schist strata and pegmatitic material.

Alteration noted in the biotite-rich schist strata, when caught within the aureole, indicates that there was always a mafic mineral stable under the conditions imposed. Metamorphism of high thermal type follows conventional lines and presents no problem.

Alteration noted in the arenaceous strata suggests that a magmoid condition was reached. There is flow or movement. This magmoid forms fairly large masses of irregular extent with uniform composition. It solidifies to the rock that has been called "pink diabase" and "granophyre."

Result of sodic vapors acting on felsic minerals can be studied to best advantage in the pegmatite veins. Where these have been caught within the aureole, residual cores of orthoclase and quartz (strained) are surrounded by zones of fine-grained micropegmatite, albite and quartz (straight).

Author's conclusions:

Micropegmatite can form from pegmatite minerals by soda pneumatolysis.

Granophyre or pink diabase, as occurring at Safe Harbor, Pa., derived from sedimentary strata composed essentially of granitic minerals by soda pneumatolysis.

FOUR SERIES OF ALKALI FELDSPARS

O. F. TUTTLE AND N. L. BOWEN
Geophysical Laboratory, Washington, D. C.

On the basis of x-ray and optical examination alkali feldspars may be divided into four series: microcline-low albite, orthoclase-low albite, sanidine-high albite, and "high" sanidine-high albite. The first three series usually occur in nature as cryptoperthite, a condition which may be revealed only by x-rays. All specimens (5) examined, which belong to the microcline-low albite series, consist of a submicroscopic intergrowth of nearly pure microcline and pure low albite. The orthoclase-low albite series is also usually unmixed to pure orthoclase and pure low albite of submicroscopic dimensions. Representatives of the sanidine-high albite series are usually completely unmixed to pure potash feldspar and pure soda feldspar; only rarely are they homogeneous. Some optically homogeneous representatives of this series consist of three phases: a potash feldspar, soda feldspar, and an intermediate phase representing the original material still unmixed. Synthetic feldspars belong to the high sanidine-high albite series. Natural alkali feldspars can be converted to homogeneous members of this series by appropriate heat treatment.

Optical properties are given for distinguishing the four series. The changes of optical properties with composition and heat treatment are discussed.

BLISTER HYPOTHESIS AND THE PETROGENIC CYCLE

C. W. WOLFE
Boston University, Boston, Massachusetts

Many writers have suggested that magmas are the direct result of radioactivity. The Blister Hypothesis would suggest that all magmas are directly or indirectly the result of this process and are derived from previously existing solid rock. Since a Blister is a relatively warmer and expanded subcrustal sector, any level within the Blister may become magma under proper conditions of pressure. It is assumed that some differentiation but almost no convection takes place in the Blister when it becomes sufficiently warmed to form a fluid phase. The particular magmatic type which develops depends principally upon the nature of the rock which is transformed into magma and upon the extent of that transformation. Above the roof of the Blister where plateau flows are apt to form, the sequence of magmatic types is thought to result from the formation of magmas at various levels in a layered crust, although some magmatic differentiation certainly obtains. Hybrid magmas result from the transformation of unusual parent rocks or from mixing during translation and emplacement of magmas from non-homogeneous sources. Geosynclinal sectors are apt to yield rather pure femag pretectonic magmas, variable but usually silicic concordant tectonic magmas derived from the transformation of sediments, and hybrid discordant post-tectonic magmas. The late post-tectonic magmas are apt to be of deep-seated origin and femag in character. It is assumed that the presence of Blister makes such petrogenesis possible, not only through its provision of heat but also through its provision of forces sufficient for orogenesis.

STABILITY RELATIONS OF CLINOCHLORE AND CORDIERITE IN THE SYSTEM MgO-Al_2O_3-SiO_2-H_2O

HATTEN S. YODER, JR.
Geophysical Laboratory, Washington, D. C.

Equilibria in a portion of the system MgO-Al_2O_3-SiO_2-H_2O have been determined at temperatures from 450° to 900° C. at pressures of water vapor up to 30,000 psi. The univariant pressure-temperature curves have been bracketed for the following reactions:

$$\text{Clinochlore} \rightleftarrows \text{forsterite} + \text{cordierite} + \text{spinel} + \text{vapor},$$
$$\text{Talc} + \text{clinochlore} \rightleftarrows \text{forsterite} + \text{cordierite} + \text{vapor},$$

and the reactions in the MgO-SiO_2-H_2O system investigated by Bowen and Tuttle (1949) are confirmed.

Pure clinochlore has a maximum stability limit of 680° C. at 15,000 psi water vapor pressure. Below about 550° C. a new form having the clinochlore composition is obtained. It is suspected that this form, although its stability has not been fixed with certainty, may be isostructural with one form of serpentine.

Cordierite is stable at least as low as 510° C. in the presence of water vapor. Pyrope was not synthesized and it is concluded that it is not stable in the presence of an excess of water vapor in the temperature range investigated. Amesite and magnesian chamosite were not produced. The metastable formation of the phases mentioned above is common and their reaction rates are very slow.

It can be demonstrated that several common hydrothermal alterations which have been interpreted as retrograde metamorphism, in the sense of lower grade, need not mean a change in pressure or temperature conditions, but may mean access of water vapor. The presence of an "excess" or "deficiency" of water vapor greatly influences the mineralogy of a metamorphic rock.

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1950 Clifford Frondel, Lewis S. Ramsdell, E. F. Osborn.
1951 Cliford Frondel, Lewis S. Ramsdell, E. F. Osborn, George T. Faust.

Annual Meeting Places

1920 Chicago, Illinois
1921 Amherst, Massachusetts
1922 Ann Arbor, Michigan
1923 Washington, D. C.
1924 Ithaca, New York
1925 New Haven, Connecticut
1926 Madison Wisconsin
1927 Cleveland, Ohio
1928 New York, N. Y.
1929 Washington, D. C.
1930 Toronto, Canada
1931 Tulsa, Oklahoma
1932 Cambridge, Massachusetts
1933 Chicago, Illinois
1934 Rochester, New York
1935 New York, N. Y.
1936 Cincinnati, Ohio
1937 Washington, D. C.
1938 New York, N. Y.
1939 Minneapolis, Minnesota
1940 Austin, Texas
1941 Boston, Massachusetts
1942 No meeting held
1943 No meeting held
1944 No meeting held
1945 Pittsburgh, Pennsylvania
1946 Chicago, Illinois
1947 Ottawa, Canada
1948 New York, N. Y.
1949 El Paso, Texas
1950 Washington, D. C.
1951 Detroit, Michigan

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CORRESPONDENTS

Eskola, Pentti, Geological Institute of the University, Snellmaninkatu 5, Helsinki, Finland.

Niggli, Paul, Eidg. Technische Hochschule, Min.-Pedro. Institut, Sonneggestrasse 5, Zürich, Switzerland.

Slavík, Frantisek, Albertov 6, Praha, Czechoslovakia.

Spencer, L. J., 111 Albert Bridge Road, London, S.W. 11, England.

Tilley, C. E., Cambridge University, Cambridge, England.

FELLOWS AND MEMBERS

(* Indicates Fellow)

Adams, John Wagstaff, Box 2767, Lakewood Branch, Denver 15, Colorado.

Adler, Hans H., 34 Ketcham Avenue, Hicksville, Long Island, New York.

Aghion, Henri, 26, Rue Chérif Pacha, Alexandrie, Le.-Egypte.

Agrell, S. O., Dept. of Geology, University of Manchester, Manchester 13, England.

*Ahlfeld, Federico, Casilla 582, Cochabamba, Bolivia.

Ahrens, L. H., Dept. of Geology, Massachusetts Institute of Technology, Cambridge 39, Massachusetts.

Albers, John P., 632 Fairmede Avenue, Palo Alto, California.

Albrecht, Herbert O., 62 S. Hillcrest Road, Springfield, Pennsylvania.

Aldrich, Dean C., 11 Buena Vista, Mill Valley, California.

*Aldrich, Henry R., 200 Van Orden Avenue, Leonia, New Jersey.

Alessio, Oreste G., Jr., 3831 Alta Vista Terrace, Chicago 13, Illinois.

Alexander, A. E., 55 East 47th Street, New York, New York.

Alf, Raymond M., Webb School, Claremont, California.

Allen, Alfred W., Dept. of Ceramic Engineering, University of Illinois, Urbana, Illinois.

Allen, C. M., Bay House, Pembroke, Bermuda.

Allen, Robert D., 32 Tyson Road, Worcester 6, Massachusetts.

*Allen, Victor T., Institute of Geophysical Technology, 3621 Olive Street, St. Louis 8, Missouri.

*Alling, Harold L., 155 West Jefferson Road, Pittsford, New Jersey.

*Allison, C. A., c/o Milligan Beauty Parlor, Miles City, Montana.

Altschuler, Z. S., U. S. Geological Survey, Geochemisty & Petrology, Washington 25, D. C.

Anderson, A. Benton, Hazardville, Connecticut.

*Anderson, Alfred L., Dept. of Geology, Cornell University, Ithaca, New York.

*Anderson, Charles A., Box 633, Prescott, Arizona.

Anderson, J. L., c/o Ralph E. Davies, Commerce Building, Houston, Texas.

Andreatta, Ciro, Istituto di Mineralogia e Petrograffia, Universita di Bologna, Via Zamboni 94, Bologna, Italia.

Andrew, Gerald, Box 410, Geological Survey, Khartum, Anglo-Egyptian Sudan.

Andrews, T. G., Geology Department, Box 2005, University, Alabama.

Angove, John, c/o Mysore Gold Mining Co., Ltd., Marikuppuppan P.O., Mysore, South India.

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Anthony, John W., Dept. of Geology, University of Arizona, Tucson, Arizona.
Arellano, A. R. V., Apartado 8393, Mexico, D.F., Mexico.
Arney, Tom, 110 West 2nd Street, Newberg, Oregon.
Arnott, Ronald J., John Jay Hall, Columbia University, New York 27, New York.
Ashby, George E., Gambrills, Maryland.
Axelrod, Joseph, U. S. Geological Survey, Washington 25, D. C.
Aycardo, Man M., Jr., 596 Uito Cruz, Manila, Philippine Islands.

Bacon, Charles S., Jr., Dept. of Geology and Mineralogy, Case Institute of Technology, Cleveland 6, Ohio.
Bacon, James F., Development & Research Dept., Corning Glass Works, Corning, New York.
Bailey, Edgar H., 257 Oakdale Way, Redwood City, California.
Bailey, R. K., 1725 Taylor Street, N.W., Washington 11, D. C.
Bailey, S. W., Dept. of Geology, University of Wisconsin, Madison, Wisconsin.
Bailey, René, Universite de Liège, Institut de Mineralogie, Liège, Belgium.
*Bain, George W., 12 Dana Street, Amherst, Massachusetts.
Balke, Claire C., 501 Lombardy Road, Drexel Hill, Pennsylvania.
Ballesteros, Alfonso, Surigao Mineral Acres, Lanuza, Surigao, Philippines.
*Bandy, Mark C., Redfield, Iowa.
Bank, Walter, U. S. Bureau of Mines, 4800 Forbes Street, Pittsburgh 13, Pennsylvania.
*Bannerman, Harold M., U. S. Geological Survey, Washington 25, D. C.
*Bannister, F. A., 34 Monahan Avenue, Purley, Surrey, England.
*Barksdale, Julian D., Dept. of Geology, University of Washington, Seattle 5, Washington.
*Barlett, Helen Blair, AC Spark Plug Division, General Motors Corporation, Flint 2, Michigan.
*Barnes, Virgil E., 207 East 33rd Street, Austin 21, Texas.
*Barnes, W. C., 215 Regent Street, Glen Ellyn, Illinois.
*Barnes, William H., Radiology Section, National Research Council, Ottawa, Canada.
Barrett, Richard L., 455 El Prado, Las Cruces, New Mexico.
*Barth, Tom F. W., Mineralogisk Geologisk Museum, Oslo 45, Norway.
Bassett, Allen M., 1985 Cody Street, Lakewood, Colorado.
*Bateman, Alan M., Dept. of Geology, Yale University, New Haven, Connecticut.
*Bates, Thomas F., Dept. of Earth Sciences, Pennsylvania State College, State College, Pennsylvania.
*Bauer, Lawson H., Franklin, New Jersey.
Baumann, H. N., Research Dept., The Carborundum Company, Niagara Falls, New York.
Baxter, E. F., Calle Viamonte 470, Cordoba, Argentina.
Beal, I. A., Ruby Star Route, Box 38, Tucson, Arizona.
Beard, E. H., The Imperial Institute, London, S.W. 7, England.
Beck, Carl W., Dept. of Geology, University of New Mexico, Albuquerque, New Mexico.
Beck, Warren, Central Research Dept., Minnesota Mining & Manufacturing Company, 367 Grove Street, St. Paul, Minnesota.
*Behre, Charles H., Jr., Dept. of Geology, Columbia University, New York 27, New York.
Bell, Gordon L., 1520 Kensington Avenue, Salt Lake City 5, Utah.
*Bell, James Forbes, 1510 Public Service Building, Portland 4, Oregon.
Benthin, B. M., Tenino, Washington.

Berkelhammer, Louis H., Ohmite Manufacturing Company, 4835 Flournoy Street, Chicago 44, Illinois.
Berman, Joseph, U. S. Geological Survey, Naval Gun Factory, Washington, D. C.
Bernheimer, Alan W., Dept. of Bacteriology, N. Y. U. College of Medicine, 477 1st Avenue, New York, New York.
*Berry, Leonard G., Dept. of Mineralogy, Queen's University, Kingston, Ontario, Canada.
Bever, James E., Mineralogical Laboratory, University of Michigan, Ann Arbor, Michigan.
Biggs, Donald L., 1208 Paquin Avenue, Columbia, Missouri.
*Billings, Marland P., Dept. of Geology, Harvard University, Cambridge 38, Massachusetts.
Bingham, E. Burris, 8436 Ramsgate, Los Angeles 45, California.
Birchall, Benjamin H., 234 East Penn Street, Philadelphia 44, Pennsylvania.
Birchman, Paul, 717 East Randolph Street, Angola, Indiana.
Biren, Mrs. H. A., 138-10 Cronston Avenue, Belle Harbor, New York.
Bjareby, Gunnar, 147 Worthington Street, Boston 15, Massachusetts.
Blank, Horace R., Dept. of Geology, Texas A. & M. College, College Station, Texas.
Blois, Everett C., Jr., Summer Lane, Framingham, Massachusetts.
Blondel, Fernand A., 12 Rue de Bourgogne, Paris VII, France.
Bloom, Joseph B., 7810 Brewster Avenue, Philadelphia 42, Pennsylvania.
Bloss, F. Donald, Dept. of Geology, University of Tennessee, Knoxville, Tennessee.
Boase, Woody Carl, 109 Main Street, Rapid City, South Dakota.
Bogue, Richard G., 2943 E. 31st Street, Tucson, Arizona.
Booth, Oliver E., 907 Clinton Avenue, Des Moines 13, Iowa.
Borgstrom, L. H., Mussigatan 3, Helsingfors, Finland.
Botinelly, Theodore, 4807 41st Street, N.W., Washington, D. C.
Boucot, Arthur J., Apt. 2, 4000 North Fifth Street, Arlington, Virginia.
*Bowen, Norman L., Geophysical Laboratory, 2801 Upton Street, N.W., Washington 8, D. C.
Bowie, Stanley H. U., Geological Survey of Great Britain, Exhibition Road, London S.W. 7, England.
*Bowles, Oliver, 5000 Massachusetts Avenue, N.W., Washington 8, D. C.
Boynton, A. G., Box 611, Boulder City, Nevada.
Brace, William F., 319 Highland Avenue, Winchester, Massachusetts.
Bradley, J. W., 4300 Budlong Avenue, Los Angeles 37, California.
*Bradley, William F., Illinois State Geological Survey, Urbana, Illinois.
*Bramlette, Milton N., Scripps Institute of Oceanography, La Jolla, California.
Brant, Arthur M., Lord Hall, Ohio State University, Columbus, Ohio.
Bray, D. M., 42 William Street, Roseville, New South Wales, Australia.
Brecke, Ervin A., Rosiclare, Illinois.
Brendler, W., Winterstrasse 4–8, Hamburg-Altona, Germany.
Brineman, John H., 2800 Northwest Highway, Dallas 9, Texas.
Brison, R. J., 215 West Lakeview Avenue, Columbus, Ohio.
Brobst, Donald A., U. S. Geological Survey, Room 13, Post Office Building, Knoxville, Tennessee.
Britton, Marvin G., Dept. of Mineralogy, Ohio State University, Columbus 10, Oho.
Brown, Arthur H., R. F. D. Mission Road, Jessups, Maryland.
*Brown, John S., c/o St. Joseph Lead Company, Bonne Terre, Missouri.
Brown, Truehart, Geochemical Survey, 3806 Cedar Springs, Dallas, Texas.

BROWN, T. R., Dept. of Geology, University of Kentucky, Lexington 29, Kentucky.
BROWNELL, WAYNE E., 126 Hornell Street, Hornell, New York.
BRUNK, C. C., 2809½ West 42nd Street, Los Angeles 8, California.
BRYANT, W. M. D., R.D. 3, Silverside Road, Wilmington, Delaware.
BUCKELEW, HAROLD C., 24 Lyons Avenue, Newark 8, New Jersey.
*BUCKLEY, HAROLD E., 100 Hazelhurst Road, Worsley, Manchester, England.
BUCKLEY, S. E., Humble Oil & Refining Company, P.O. Box 2180, Houston 1, Texas.
*BUDDINGTON, ARTHUR F., Dept. of Geology, Princeton University, Princeton, New Jersey
*BUERGER, MARTIN J., Massachusetts Institute of Technology, Cambridge 39, Massachusetts.
*BUERGER, NEWTON W., Dept. of Metallurgy, U. S. Naval Academy, Annapolis, Maryland.
*BULLARD, FRED M., Dept. of Geology, University of Texas, Austin, Texas.
BULLOCK, KENNETH C., Dept. of Geology, Brigham Young University, Provo, Utah.
BUNTON, PAUL B., 2900 Connecticut Avenue, N.W., Washington 8, D. C.
*BURBANK, WILBUR S., 3348 Runnymede Place, N.W., Washington 15, D. C.
*BURFOOT, JAMES DABNEY, JR., Dept. of Geology, Cornell University, Ithaca, New York.
BURNHAM, C. WAYNE, 2385 Oswego Street, Pasadena 10, California.
BURNHAM, G. E., 128½ S. Encinitas Avenue, Monrovia, California.
BURRI, CONRAD, Mineralogisch-petrographisches Institut, Eidgenössische Technische Hochschule, Sonneggstrasse 5, Zürich 6, Switzerland.
BURST, JOHN F., 3737 Bellaire Boulevard, Shell Oil Company, Houston, Texas.
BUTLER, E. S., 408 Archer Street, Houston 9, Texas.
*BUTLER, G. MONTAGUE, Box 4186, University Station, Tucson, Arizona.
BUTLER, JOHN W., JR., c/o Phillips Venezuelan Oil Company, Apartado 1031, Caracas, Venezuela.
*BUTLER, ROBERT D., Bethlehem Steel Company, Bethlehem, Pennsylvania.
*BUTTGENBACH, HENRY J., 182 Avenue F. D. Roosevelt, Bruxelles, Belgium.
BYERS, FRANK M., JR., General Geology Branch, Federal Center, Denver 14, Colorado.
BYRON, J. E., 1240 Pearl Street, Boulder, Colorado.

CALLAHAN, WILLIAM H., 1 Rowe Place, Franklin, New Jersey.
CALVER, JAMES L., 1553 Cristobal Drive, Los Robles, Tallahassee, Florida.
CALVERT, EARL L., 1528 Ardendale Avenue, San Gabriel, California.
*CAMERON, EUGENE N., Science Hall, Madison 6, Wisconsin.
*CAMPBELL, CHARLES D., Harvey Road, Pullman, Washington.
*CAMPBELL, IAN, California Institute of Technology, Pasadena 4, California.
CANAL, PAUL, 4 Place Bir-Hacheim, Rueil-Malmaison (S&O), France.
*CANNON, RALPH S., JR., U. S. Geological Survey, Washington 25, D. C.
*CAPLAN, ALLAN, 2 West 46th Street, New York 19, New York.
CARDOSO, GABRIEL M., Andres Mellado 7, 3rd isgda, Madrid, Spain.
CARGILLE, R. P., 118 Liberty Street, New York 6, New York.
CARL, HOWARD F., Highland, Howard County, Maryland.
*CAROBBI, GUIDO, Istituto de Mineralogia della Universita, Piassa S. Marco L., Florence, Italy.
CARTER, JOE O., Apartado 234, Maracaibo, Venezuela.
CASPERSON, WILLIAM C., Paterson Museum, 268 Summer Street, Paterson, New Jersey.
CASTILLO, JUAN S., Ayungan, Negros Oriental, Philippines.
CHANG, K. H., 969 River Road Dormitories, Ohio State University, Columbus 10, Ohio.
CHANG, PING HSI, c/o Arthur Montgomery, 277 Park Avenue, New York 17, New York.

*Chapman, Carleton A., Dept. of Geology, University of Illinois, Urbana, Illinois.
*Chapman, Randolph, Dept. of Geology, Johns Hopkins University, Baltimore 18, Maryland.
Chase, Gerald W., Oklahoma Geological Survey, Norman, Oklahoma.
*Chayes, Felix, Geophysical Laboratory, 2801 Upton Street, N.W., Washington 8, D. C.
Chesley, Frank G., Central Research Laboratories, Inc., Red Wing, Minnesota.
Chesley, Richard B., 50 Pratt Street, Reading, Massachusetts.
Chesterman, Charles W., 1066 Keith Avenue, Berkeley, California.
Chidester, Alfred H., 7 Winter Street, Montpelier, Vermont.
Christ, C. L., U. S. Geological Survey, Geochemistry & Petrology, Washington 25, D. C.
Chute, Newton E., Dept. of Geology, Syracuse University, Syracuse, New York.
Clabaugh, Stephen E., Dept. of Geology, University of Texas, Suatin 12, Texas.
Claffy, Mrs. Esther W., Naval Research Laboratory, Crystal Section, Building 3, Washington 25, D. C.
*Claringbull, G. F., Dept. of Mineralogy, British Museum, Cromwell Road, London, S.W. 7, England.
Clausen, Hans, Mineralogical Museum, Oster Voldgade 7, Copenhagen, K., Denmark.
Clemens, Leland P., 8304 Bevan Street, San Gabriel, California.
Cline, Stanley H., North Road, Warren MC 54, Ohio.
*Coats, Robert, Alaskan Branch, Geological Survey, Washington 25, D. C.
*Colburn, Burnham S., Greystone Court, Biltmore, North Carolina.
*Colburn, William B., P.O. Box 456, Rancho Santa Fe, California.
Cole, G. Thurston, Rumford Point, Maine.
Collini, Fil. Lic. Bengt, St. Goransgatan 20, Uppsala, Sweden.
Comeforo, Jay Eugene, 86 Pine Road, Box 141, Norris, Tennessee.
Cook, Mrs. Kathryn H., 493 South Firestone Boulevard, Akron, Ohio.
Cook, Roger H., 1213 12 Street, Golden, Colorado.
Cooke, H. R., Jr., Northern Peru Mining & Smelting Company, Casilla 162, Trujillo, Peru.
*Cooke, S. R. B., Minnesota School of Mines, Minneapolis 14, Minnesota.
Cooley, Mary E., 703 South Forest Avenue, Ann Arbor, Michigan.
Coombs, D. S., Emmanuel College, Cambridge, England.
Cooney, Robert L., 4624 West 17 St., Los Angeles 19, California.
*Cooper, John R., U. S. Geological Survey, Washington 25, D. C.
*Corbett, Clifton S., 1102 Talon Avenue, Calgary, Alta., Canada.
*Correns, Carl W., Lotzestrasse 13, Göttingen, Germany, British Zone.
Corwin, Gilbert, 2122A Folwell Avenue, St. Paul 8, Minnesota.
Cotter, Perry G., 24 East Circle Road, Norris, Tennessee.
Cox, W. R., 2114 East Burnside, Portland, Oregon.
Crawford, Arthur L., 1067 East Fifth Street, S., Salt Lake City, Utah.
Creitz, Ellis E., Drawer L, University, Alabama.
Cribbs, Richard E., Cobalt, Idaho.
Crippen, Richard A., Jr., Division of Mines, Ferry Building, San Francisco, California.
Crump, Robert M., Box 88, Star Lake, New York.
Crunden, A. B., 30 Porter Place, Montclair, New Jersey.
Cuervo, Dra. America Ana, Laboratorio No. 1 de Mineralogia, Escuela de Ciencias, Universidad de la Havana, Havana, Cuba.
Cummins, A. B., Cedar Crest Road, Bound Brook, New Jersey.
Cunningham, Donald E., 2719 Reel Street, Harrisburg, Pennsylvania.
*Currier, Louis W., U. S. Geological Survey, Washington 25, D. C.
Cuthbert, F., P.O. Box 1675, Houston 1, Texas.

Daggett, E. B., c/o Vanadium Corporation of America, Naturita, Colorado.
Dake, H. C., 329 S.E. 32nd Avenue, Portland 15, Oregon.
Danner, Wilbert R., Dept. of Geology, College of Wooster, Wooster, Ohio.
Darneal, Robert L., Box 832, Stanford University, California.
Da Silva, Alves, Casilla Correo 81, San Rafael, F. C. P., Argentina.
Daves, Delmer Lawrence, 107 N. Bentley Avenue, West Los Angeles 24, California.
Davey, W. P., Osmond Laboratory, Pennsylvania State College, State College, Pennsylvania.
Davidson, Mr., 9 Castlegate, Penrith, Cumberland, England.
*Davidson, Charles Findlay, Geological Survey and Museum, London, S.W. 7, England.
Davidson, Stanley C., 18 Kilbarry Road, Toronto 12, Ontario, Canada
Davis, Donald W., R.F.D. 1, Katonah, New York.
*Davis, Gordon Leslie, 4115 Wisconsin Avenue, N.W. Washington 8, D. C.
Davis, Harry T., Curator of Geology, North Carolina State Museum, Raleigh, North Carolina.
*De Bretteville, A. P., Jr., 224 Navesink Avenue, Highlands, New Jersey.
De Carlo, Charles, 2116 Walnut Street, Philadelphia 3, Pennsylvania.
de las Casas, Fernando, Geological Dept., Cerro de Pasco Copper Corporation, Cerro de Pasco, Peru.
de Lima, Plinio, Rua Virotino Carmillo 565, Sao Paulo, Brazil.
Dengo, Gabriel, Apartado 1851, San Jose, Costa Rica, Central America.
Denning, Reynolds M., 1015 E. Houghton Avenue, Houghton, Michigan.
De Sanctis, Mrs. Helen V. E., 86-16 60th Road, Elmhurst, Long Island, New York.
Desmond, Thomas C., 94 Broadway, Newburgh, New York.
Deul, Maurice, U. S. Geological Survey, Section of Geochemistry, Washington 25, D. C.
*de Villiers, Jacob E., Geological Survey, Box 401, Pretoria, South Africa.
DeVore, George W., Box 180, Laramie, Wyoming.
Dieterich, J. F., 3724 Vantage Avenue, North Hollywood, California.
Dietz, Ralph W., 108 B Byrnes, China Lake, California.
Dietz, Robert Sinclair, Navy Electronics Laboratory, San Diego 52, California.
Dodge, Nelson B., United States Steel Company, Research & Development Laboratory, 234 Atwood Street, Pittsburgh 13, Pennsylvania.
*Dodge, Theodore A., 635 North Third Avenue, Tucson, Arizona.
Doeglas, D. J., Laboratorium voor Mineralogie en Geologie, Duivendaal 2, Wageningen, Holland.
Dolar-Mantuani, L., 5911 Clement Road, Vancouver, British Columbia, Canada.
*Donnay, Mrs. Gabrielle, Dept. of Geology, Johns Hopkins University, Baltimore 18, Maryland.
*Donnay, Joseph D. H., Dept. of Geology, Johns Hopkins University, Baltimore 18, Maryland.
Doss, Carson L., P.O. Box 541, Golden, Colorado.
Dosse, A. F., Route 2, Box 66, F, Fontana, California.
Draeger, R. H., Capt. (MC), U.S.N., Naval Medical Research Institute, Bethesda 14, Maryland.
Dragsdorf, R. Dean, Dept. of Physics, Kansas State College, Manhattan, Kansas.
Draisin, Wilburt M., 21 Parmenter Terrace, West Newton 65, Massachusetts.
*Dreyer, Robert M., Dept. of Geology, University of Kansas, Lawrence, Kansas.
DuCloux, Mr., Netherlands Embassy, 117 Park Street, London W 1, England.
Duersmith, L. J., 405 Poplar Street, Columbia, Pennsylvania.
Duffendack, O. S., Philips Laboratories, Inc., Irvington-on-Hudson, New York.

Duhovnik, J. M., Dept. of Mineralogy, University, Ljubljana, Jugoslavia.
Duplin, V. J., Jr., The Babcock & Wilcox Company, Alliance, Ohio.
DuPont, James M., Myersville Road, Chatham, New Jersey.
Dutra, Kleber de Almeida, Rua Gonclaves Dias, 1860, Belo Horizonte, Brazil.
Dwornik, Edward J., 3611 62d Avenue, Landover, Maryland.

Earhart, Helen S., 820 Defoe Street, Missoula, Montana.
Earley, J. W., P.O. Drawer 2038, Pittsburgh 30, Pennsylvania.
*Ebbutt, Frank, 173 Glenrose Avenue, Toronto 5, Canada.
Eberlein, G. Donald, Box 372, Los Gatos, California.
Ebert, Heinz, Geologue, Avenida Pasteur 404, Commissao Nacional de Producao Mineral, Rio de Janeiro, Brazil.
*Eckel, Edwin B., U. S. Geological Survey, Denver Federal Center, Denver 14, Colorado.
*Eckermann, Harry von, Edeby, Sparreholm, Sweden.
Eddy, C. N., National State Bank, Boulder, Colorado.
*Edwards, Austin Burton, Geology Department, University of Melbourne, Melbourne, Australia.
Ehrmann, Martin L., 617 North Roxbury Drive, Beverly Hills, California.
Eisenberg, Marvin, Armour Research Foundation, Technology Center, Chicago 16, Illinois.
*Eitel, Wilhelm H. J., P.O. Box 348, Norris, Tennessee.
Elder, Curtis H., 101 Westwood, Columbia, Missouri.
*Ellestad, Reuben B., 712 South Cedar Lake Road, Minneapolis, Maryland.
*Ellsworth, H. V., Canada Geological Survey, Ottawa, Canada.
Elwell, Wilbur J., 2 Duck Street, Danbury, Connecticut.
Emke, Harvey J., 200 Bulman Block, 218 Bannatyne Avenue, Winnipeg, Manitoba, Canada.
Emmons, Richard C., Science Hall, University of Wisconsin, Madison, Wisconsin.
*Engel, Albert E. J., Dept. of Geology, California Institute of Technology, Pasadena 4, California.
Engel, Rene, 711 South Fair Oaks Avenue, Pasadena 2, California.
Enlows, Harold E., The University of Tulsa, Seventh and College, Tulsa 4, Oklahoma.
Erdmann, Charles E., c/o U. S. Geological Survey, P.O. Box 1827, Great Falls, Montana.
Ernst, T., Mineralogisches Institut der Universität Erlangen, Schlossgarten 5, Erlangen, Germany.
Evanof, Philip, 1842 Butler Pike, Conshohocken, Pennsylvania.
*Evans, Howard T., Jr., Philips Laboratories, Inc., Irvington-on-Hudson, New York.

Fackler, William C., Route 3, Grimm Road, Newburgh, Indiana.
*Faessler, Carl, Université Laval, Quebec, Canada.
*Fahey, Joseph J., U. S. Geological Survey, Washington 25, D. C.
Faick, John N., 825 North Second Avenue, Tucson, Arizona.
*Fairbairn, Harold W., Dept. of Geology, Massachusetts Institute of Technology, Cambridge 39, Massachusetts.
*Fankuchen, I., Polytechnic Institute of Brooklyn, Brooklyn, New York.
Farwell, Fred W., Box 328, Noroton, Connecticut.
*Faust, George T., 9907 Capitol View Avenue, Silver Spring, Maryland.
*Ferguson, R. B., Dept. of Geology, University of Manitoba, Winnipeg, Canada.
*Fettke, Charles R., 1118 Wightman Street, Squirrel Hill Station, Pittsburgh, Pennsylvania.

Filer, Alvin V., Jr., 1408 5th Avenue West, Bush Hills, Birmingham, Alabama.
Finch, James, Dominion Laboratory, Sydney Street W., Wellington N. 1, New Zealand.
Finlay, G. R., c/o Norton Company, Chippawa, Ontario, Canada.
Finnegan, Michael J., Box 289, St. Francis, Kansas.
*Fisher, D. Jerome, Dept. of Geology, University of Chicago, Chicago 37, Illinois.
Fisher, Robert B., 1107 Rolla Street, Rolla, Missouri.
Fisk, Henry G, University of Wyoming, Laramie, Wyoming.
Flack, Wylie H., 333 Borbeck Street, Philadelphia 11, Pennsylvania.
Flagg, A. L., Box 2345, Phoenix, Arizona.
Fleener, F. L., 1415 Hosmer Street, Joliet, Illinois.
Fleischer, Arthur, Box 349, Easthampton, Massachusetts.
*Fleischer, Michael, U. S. Geological Survey, Washington 25, D. C.
*Ford, Hugh Alexander, 736 Riverside Drive, Apt. 6-C, New York 31, New York.
Forman, S. A., Ceramic Section, Mines Branch, Ottawa, Canada.
*Foshag, William F., U. S. National Museum, Washington, D. C.
Foster, Margaret D., U. S. Geological Survey, Washington 25, D. C.
Foster, Philip C., R.D. 1, South Berwick, Maine.
Foster, Wilder D., R.D. 1, Box 377, Verona, Pennsylvania.
*Foster, Wilfrid Raymond, Champion Spark Plug Company, 8525 Butler Avenue, Detroit 11, Michigan.
Fox, Portland P., Caixa Postal 8026, Sao Paula Trammay Light & Power Company, Ltd., Sao Paulo, Brazil.
*Franco, Rui Ribeiro, University of Sao Paulo, Sao Paulo, Brazil.
*Fraser, Donald McCoy, Geological Department, Bethlehem Steel Company, Bethlehem, Pennsylvania.
*Fraser, Horace J., 261 Dawlish Avenue, Toronto, Ontario, Canada.
Fréchette, V. D., New York State College of Ceramics, Alfred, New York.
Freed, Olyn, 509 Evans Avenue, Reno, Nevada.
Freeze, Arthur C., Consolidated Mining and Smelting Company, Kimberley, British Columbia, Canada.
French, Irwin H., Danforth, Maine.
Fretz, A. Henry, 42 West Market Street, Bethlehem, Pennsylvania.
Fries, Carl Jr., U. S. Geological Survey, Washington 25, D. C.
Frimel, Hobart F., 32236 Annapolis Avenue, Wayne, Michigan.
*Frondel, Clifford, 12 Geological Museum, Oxford Street, Cambridge 38, Massachusetts.
Frueh, Alfred J., Jr., Dept. of Geology, University of Chicago, Chicago 37, Illinois.
*Fuller, Richard E., 1642 Federal Avenue, Seattle, Washington.
Fumi, Fausto, Institute of Theoretical Physics, University of Milan, Milan, Italy.
Fuog, H. L., 4149 Barrett Road, Los Angeles 32, California.
*Furnival, George M., c/o The California Standard Company, 200 Greyhound Building, Calgary, Alberta, Canada.

*Gabriel, Alton, U. S. Bureau of Mines, Norris, Tennessee.
*Galbraith, F. W., Dept. of Geology, University of Arizona, Tucson, Arizona.
*Gallagher, David, U. S. Geological Survey, Joplin, Missouri.
Gallitelli, Paolo, Istituto Mineralogia della Universita, Modena, Italy.
Galloni, Ing. Ernesto, Yerbal 1763, Buenos Aires, Argentina.
*Garrido, J., Lagasca 123, Madrid, Spain.
Gates, Robert M., Science Hall, Madison, Wisconsin.

*Gaudin, A. M., Room 8-211, Massachusetts Institute of Technology, Cambridge 39, Massachusetts.
Gault, H. Richard, Dept. of Geology, Lehigh University, Bethlehem, Pennsylvania.
Gaywood, Roger M., U. S. Atomic Energy Commission, P.O. Box 81, Marysvale, Utah.
Georgalas, George C., 39A Pipinou Street, Athens, Greece.
George, D'Arcy R., 272 Lexington Street, Watertown, Massachusetts.
Gerbolini, Juan F., Box 233, Lima, Peru.
*Gianella, Vincent P., Mackay School of Mines, Reno, Nevada.
Gifford, James C., 521 East Virginia Street, Tucson, Arizona.
Gillespie, T. B., Star Route, St. Augustine, Florida.
Gillingham, Thomas E., 23 Welshman Court, Caldwell, New Jersey.
*Gillson, Joseph L., 8058 DuPont Building, Wilmington, Delaware.
*Gilluly, James, U. S. Geological Survey, Denver Federal Center, Denver 14, Colorado.
Girault, J. P., 16, St. Denis Avenue, Quebec City, Quebec, Canada.
Girhard, M. Nancy, U. S. Geological Survey, Geochemistry & Petrology, Washington 25, D. C.
Givens, David B., 3417 Monte Vista, Albuquerque, New Mexico.
Glass, Herbert D., Illinois State Geological Survey, Urbana, Illinois.
*Glass, Jewell, J., U. S. Geological Survey, Washington 25, D. C.
Godat, E. A., Apartado 251, Monterrey, Mexico, c/o Cia. Minera de Penoles.
*Goddard, Edwin N., Dept. of Geology, University of Michigan, Ann Arbor, Michigan.
*Goldich, Samuel S., Dept. of Geology, University of Minnesota, Minneapolis 14, Minnesota.
*Goldsmith, J. R., Dept. of Geology, University of Chicago, Chicago 37, Illinois.
*González-Bonorina, Félix, Julian Alvarez 2430, Buenos Aires, Argentina, South America.
*Goodspeed, George E., Dept. of Geology, University of Washington, Seattle, Washington.
*Gordon, Samuel G., 4168 Trinity Drive, Los Alamos, New Mexico.
Goring, Arthur W., Box 183, Ruth, Nevada.
Gorman, D. H., 245 Sammon Avenue, Toronto, Canada.
Goudey, Hatfield, Box 401, Yerington, Nevada.
Grace, Marvin, 8162 Ogden Street, Houston, Texas.
Graham, A. R., 1851 Yonge Street, Toronto, Ontario, Canada.
*Graham, R. P. D., McGill University, Montreal, Canada.
Grahl, Harry, 885 Cauldwell Avenue, Bronx, New York.
*Graton, Louis C., Vanserg Laboratories, Cambridge 38, Massachusetts.
*Grawe, Oliver R., Dept. of Geology, Missouri School of Mines, Rolla, Missouri.
Green, Robert S., 2501 Onondaga Drive, Columbus 12, Ohio.
Greene, Kenneth T., 746 Gaylord Street, Denver 6, Colorado.
Gregory, Robert F., 10892½ Strathmore Drive, Los Angeles 24, California.
Gregory, W. A., Jr., 59 First Street, New Britain, Connecticut.
*Greig, Joseph W., Geophysical Laboratory, 2801 Upton Street, N.W., Washington 8, D.C.
Griffiths, John Cedric, Dept. of Earth Sciences, Pennsylvania State College, State College, Pennsylvania.
Griffitts, Wallace R., P.O. Box 236, Shelby, North Carolina.
Griggs, Roy L., U. S. Geological Survey, P.O. Box 443, Albuquerque, New Mexico.
*Grim, Ralph E., 234 Natural History Building, University of Illinois, Urbana, Illinois.
Grogan, Robert M., Room 8058, DuPont Building, Wilmington, Delaware.

Gross, Eugene B., 755 15th Street, Boulder, Colorado.
Grossman, Irving G., Box 590, U. S. Geological Survey, Albany, New York.
*Grout, Frank F., Dept. of Geology, University of Minnesota, Minneapolis, Minnesota.
Gruey, Merle N., 189 Vine, Geneva, Ohio.
*Gruner, John W., Dept. of Geology, University of Minnesota, Minneapolis, Minnesota.
Grunig, J. K., Mineral Deposits Branch, U. S. Geological Survey, Washington 25, D. C.
Gude, Arthur James, III, Box 374, Golden, Colorado.
Guillemin, M., 265 rue Lecourbe, Paris 15, France.
Guimaraes, Djalma, Rua Goncalves Dias, 3182, Belo Horizonte, Brazil.
*Gummer, Wilfrid K., 815 Fifth Street, Arvida, Quebec, Canada.
Gunnell, E. M., 3365 East Kentucky Avenue, Denver, Colorado.
Gush, Mrs. I. N., Box 1167, Johannesburg, South Africa.
*Gysin, M. E., Laboratoire de Minéralogie de l'Université, Quai de l'Ecole-de-Médecin, Genève, Switzerland.

Haddock, William J., 7700 Boeing Avenue, Los Angeles 45, California.
Haecker, A. J., Jr., Georgia Kaolin Company, 1185 Nary Street, Elizabeth, New Jersey.
*Haff, John C., Dept. of Geology, Mount Holyoke College, South Hadley, Massachusetts.
Hagner, Arthur F., Dept. of Geology, University of Illinois, Urbana, Illinois.
Halbouty, M. T., Suite 729–32, Shell Building, Houston 2, Texas.
Haley, Ed, Box 397, Buena Vista, Colorado.
Hall, F. P., 206 Granger Road, Syracuse 9, New York.
*Hallimond, Arthur Francis, Geological Survey and Museum, Exhibition Road, South Kensington, London, S.W. 7, England.
Hallinan, John M., Dept. of Geology, University of Sydney, New South Wales, Australia.
Hallowell, Horace J., 323 Main Street, Danbury, Connecticut.
Halsey, J. H., Bacon Hill, University of California, Berkeley, California.
Ham, William Eugene, State Geological Survey, Norman, Oklahoma.
Hanley, John B., Section of Non-metalliferous Geology, U. S. Geological Survey, Washington 25, D. C.
*Harcourt, George Alan, Copper Cliff, Ontario, Canada.
Hardy, John A., RFD 1, Salisbury, Connecticut.
Harrar, J. G., The Rockefeller Foundation, Calle de Londres 45, Mexico, D.F., Mexico.
Hartman, Robert F., 1107 16th Street, Golden, Colorado.
Hassan, N., Room 120, Soils Division, Hilgard Hall, University of California, Berkeley 4, California.
*Hatch, Robert A., U. S. Bureau of Mines, Norris, Tennessee.
*Hawkins, A. C., 826 First Place, Plainfield, New Jersey.
Hawley, F. G., 1624 E. Fifth Street, Tucson, Arizona.
*Hawley, James E., Dept. of Mineralogy, Queen's University, Kingston, Ontario, Canada.
*Haycock, Maurice Hall, 525 Broadview Avenue, Ottawa, Canada.
Hayes, Julian Lee, P.O. Box 751, Park City, Utah.
Hayes, Junius J., 1148 East 1st S., Salt Lake City 2, Utah.
Hearn, Jane K., 39 DeKalb Place, Morristown, New Jersey.
Hearne, L. F., Box 589, Wheeling, West Virginia.
Heineman, Robert E. S., 20 Calle Conquista, Arizona.
Heinicke, Kurt J., P.O. Box 2872, Hollywood, Florida.
*Heinrich, E. Wm., Mineralogical Laboratory, University of Michigan, Ann Arbor, Michigan.
Henderson, Donald M., Dept. of Geology, University of Illinois, Urbana, Illinois.

*Henderson, Edward P., U. S. National Museum, Washington, D. C.
*Hendricks, Sterling B., 1118 Dale Drive, Silver Spring, Maryland.
Henry, Norman F. M., Dept. of Mineralogy and Petrology, University of Cambridge, Downing Street, Cambridge, England.
Hentschel, H. W., DeKalb Pike, Washington Square, RD3, Norristown, Pennsylvania.
Herz, Norman, American School of Classical Studies, 54 Souidias Street, Athens, Greece.
Herzenberg, Robert, Casilla 23, Oruro, Bolivia, South America.
*Hess, Frank L., 5509 Roosevelt Street, Bethesda 14, Maryland.
Hess, Harold D., 759 Monroe, Corvallis, Oregon.
*Hess, Harry H., Dept. of Geology, Princeton University, Princeton, New Jersey.
*Hewett, Donnell F., 1460 Rose Villa Street, Pasadena, California.
Hewitt, D. F., 1195 Avenue Road, Toronto, Ontario, Canada.
Heyl, Allen V., Jr., Apt. 102, 8218 14th Avenue, Hyattsville, Maryland.
*Heyl, George R., Apartado 889, Creole Petroleum Corporation, Caracas, Venezuela, South America.
Hietanen-Makela, Anna, U. S. Geological Survey, Washington 25, D. C.
Higazy, Riad A, Grant Institute of Geology, Kings Building, University of Edinburgh, Edinburgh, Scotland.
Hildebrand, Fred A., 7916 15th Street, Apt. 102, Hyattsville, Maryland.
Hill, H. Stanton, Geology Department, Pasadena City College, Pasadena 4, California.
Hill, V. D., Route 7, Box 188, Wooster, Ohio.
Hill, V. G., Reynolds Jamaica Mines, Lydford P10, St. Ann, Jamaica, British West Indies.
Hill, William L., 321 Ingraham Street, N.W., Washington 11, D. C.
Hills, Ralph L., 18 Greenwood Avenue, Hartford 12, Connecticut.
Hilmy, Mohammed E., 415 Church Street, Ann Arbor, Michigan.
Hinckley, Arthur T., 937 Harrison Avenue, Niagara Falls, New York.
Hinsey, George H., 511 29 Street, Sacramento 16, California.
Ho, T. L., Dept. of Geology, National University of Shantung, Tsingtao, China.
*Hodge, Edwin T., 2915 Luray Terrace, N.W., Portland, Oregon.
*Holden, Guerdon S., Plain Dealer Building, Cleveland, Ohio.
Holley, Kenneth E., 113 Pritchard Avenue, Corning, New York.
*Holmes, Arthur, University of Edinburgh, Grant Institute of Geology, King's Buildings, West Mains Road, Edinburgh 9, Scotland.
Holmes, Mrs. M. A., 1 West 72 Street, New York 23, New York.
*Holmes, Ralph J., Dept. of Geology, Columbia University, New York 27, New York.
Holser, William T., Dept. of Geology, McGraw Hall, Cornell University, Ithaca, New York.
Hooker, Marjorie, Ingleside Avenue, McLean, Virginia.
Hopkins, Mrs. Joan Prewitt, RFD 5, Box 334-B, Franconia, Virginia.
Horn, Aubrey E., c/o F. H. Pigeon, P.O. Box 42, Jos, Plateau Province, Northern Nigeria.
Horovitz, Th., 45 Avenue Sidi Gaber, Cleopatra les Bains, Ramleh, Alexandria, Egypt.
Howells, Lewis W., Orefield, RFD #1, Pennsylvania.
*Howland, Arthur L., Dept. of Geology, Northwestern University, Evanston, Illinois.
Hoyt, Lester H., 1604 North Capitol Avenue, Indianapolis 7, Indiana.
Hubbard, Bela, 1405 E. Kleindale Road, Tucson, Arizona.
Hufnagel, Alvin A., 1150 Carroll Street, Brooklyn 25, New York.
Hugill, William, Langham House, Fields Road, Alsager, Cheshire, England.
Hummel, Floyd A., The Pennsylvania State College, Dept. of Mineral Technology, State College, Pennsylvania.

*Hunt, Walter F., Mineralogical Laboratory, University of Michigan, Ann Arbor, Michigan.

*Hurlbut, C. S., Jr., Dept. of Mineralogy, Harvard University, Cambridge 38, Massachusetts.

Hurley, Donal, 16 Lewis Street, Little Falls, New York.

Hurst, Thomas L., Monsanto Chemical Company, Columbia, Tennessee.

*Hutton, C. Osborne, Dept. of Mineral Sciences, Stanford University, Palo Alto, California.

Ichimuru, Takeshi, Institute of Earth Science, Tokyo University, Komaba, Meguro-ku, Tokyo, Japan.

*Ingerson, Earl, U. S. Geological Survey, Washington 25, D. C.

Ingram, W. F., P.O. Box 23, 433 West Taylor Street, Griffin, Georgia.

*Insley, Herbert, National Bureau of Standards, Washington 25, D. C.

Irving, Earl M., U. S. Geological Survey, c/o American Embassy, Manila, Philippines, APO 736, c/o Postmaster, San Francisco, California.

Irwin, William H., 215 East 19 Avenue, Denver 5, Colorado.

Ishioka, Kokichi, Nagoya University at Misuho, Nagoya, Japan.

Isotoff, A., Dept. of Geology, Idaho State College, Pocatello, Idaho.

Iyer, L. A. N., Patna University, Bankipore P.O., Patna, India.

Jackson, Kern C., Geology Department, New Engineering Building, University of Maine, Orono, Maine.

Jackson, M. L., Room 104B, Soils Building, University of Wisconsin, Madison 6, Wisconsin.

*Jacobs, Elbridge C., 146 Williams Street, Burlington, Vermont.

Jacobs, Louis J., The Obermayer Company, 2563 W. 18th Street, Chicago 8, Illinois.

Jaffe, Arthur, 3212 W. Dauphin Street, Philadelphia, Pennsylvania.

*Jaffe, Howard W., 7902 14th Avenue, Hyattsville, Maryland.

*Jahns, Richard H., Division of Geological Sciences, California Institute of Technology, Pasadena 4, California.

James, C. C., Burford, Alexandra Place, Penzance, Cornwall, England.

*James, Harold L., U. S. Geological Survey, Box 15, Iron River, Michigan.

Januzzi, Ronald Everett, 83 Elm Street, Danbury, Connecticut.

Jarrell, Oscar W., 149 Broadway, New York 6, New York.

*Jeffries, Charles D., Dept. of Agronomy, Agricultural Building, State College, Pennsylvania.

Jensen, David E., 199 East Brook Road, Pittsford, New York.

Jesseman, Donald S., 186 Hillside Road, Oak Ridge, Tennessee.

Jizba, Zdenek V., 831 West Dayton, Madison, Wisconsin.

Johne, C. T., Kragerö, Norway.

Johns, William D., 9 Natural Resources Building, University of Illinois, Urbana, Illinois

Johnson, Clayton H., 102 Swallow Hall, Columbia, Missouri.

Johnson, Mrs. Helena, Johnson Hall, David Lipscomb College, Nashville 4, Tennessee.

*Johnson, J. Harlan, Colorado School of Mines, Golden, Colorado.

*Johnson, Richard, 9 Wicker Street, Ticonderoga, New York.

Johnson, Robert F., 2575 Virginia Street, Berkeley 9, California.

Johnson, Sarah C., 1424 Somerset Place, N.W., Apt. 1, Washington, D. C.

Johnson, W. M., RFD 6, Knoxville, Tennessee.

*Johnston, R. A. A., 112 Old Forest Hill Road, Toronto 10, Ontario, Canada.

Johnston, W. G., Elk Lake, Ontario, Canada.

*Johnston, William D., Jr., U. S. Geological Survey, Washington 25, D. C.
*Jolliffe, Alfred W., Dept. of Geology, Queens University, Kingston, Ontario, Canada.
Jones, Gardner H., 8 Hill Street, Lakeport, New Hampshire.
Jones, W. R., School of Mines, Dept. of Geology, Kalgoorlie, Western Australia.

Kadey, Frederic L., Jr., 181 Mercer Street, Apt. 7A, Somerville, New Jersey.
Kaiman, S., 552 Booth Street, Ottawa, Canada.
Kaley, Mary Elizabeth, California Research Corporation, Box 543, Whittier, California.
Karageorge, Elisseos D., Myriophitou Street 5, New Smyrna, Athens, Greece.
Karrer, Sebastian, c/o Milwaukee Gas Specialty Company, 730 N. Jackson Street, Milwaukee, Wisconsin.
Kasey, J. Bryant, Box 968, Bakersfield, California.
Kaspar, Jan Vhopkach 13, Praha 13, Czechoslovakia.
Kauffman, A. J. Jr., 3119 Homewood Parkway, Kensington, Maryland.
Kazazian, Avedia M., Casilla No. 240, Antofagasta, Chile.
Kedesdy, H. H., 65 Little Silver Parkway, Little Silver, New Jersey.
*Keevil, Norman Bell, 11 Jordan Street, Toronto, Ontario, Canada.
*Keith, Mackenzie L., 204 Mineral Science Building, State College, Pennsylvania.
*Keller, Walter D., 208 Swallow Hall, Columbia, Missouri.
*Kelley, Walter P., 120 Hilgard Hall, University of California, Berkeley 4, California.
Kemp, C. E., Sault Ste. Marie Branch, Michigan College of Mining & Technology, Sault St. Marie, Michigan.
*Kennedy, George C., Geological Museum, Harvard University, Cambridge 38, Massachusetts.
*Kennedy, William Q., Dept. of Geology, University of Leeds, Leeds 2, England.
Keough, M., P.O. Box 44, Que Que, Southern Rhodesia.
*Kerr, Paul F., Dept. of Geology, Columbia University, New York 27, New York.
Kessler, Charles N., No. 3 Berg Apts., Helena, Montana.
Kidwell, Albert L., Carter Oil Company, P.O. Box 801, Tulsa, Oklahoma.
Kienreich, Jos. A., Sackstrasse 6, Graz, Austria.
Kietz, Adolph, Box 1150, RFD 1, Bellevue, Washington.
King, Myrle E., 3640 Glencoe, Denver, Colorado.
Kinsolving, May Risch, 138 West 91st Street, New York, New York.
Kirkpatrick, Alan F., 150 Courtland Hill Street, Stamford, Connecticut.
Kissileff, M. Z., 1340 Kerper Street, Philadelphia 11, Pennsylvania.
*Klein, Albert A., Norton Company, Worcester, Massachusetts.
Klein, Gilbert E., 105 West Geneva Lane, Oak Ridge, Tennessee.
Klein, Ira E., c/o Geology Division, Bureau of Reclamation, Sacramento, California.
Knabe, William C., 3853 North Gratz Street, Philadelphia, Pennsylvania.
Knizek, J. O., P.O. Box 2739, Mexico DF, Mexico.
*Knopf, Adolph, Dept. of Geology, Stanford University, Stanford, California.
Knutson, Carroll F., Box 321, Ponca City, Oklahoma.
Koebel, Charles J., 9456 Grinnell Street, Detroit 13, Michigan.
Konta, Jiri, Petrology Department, Charles University, Prague, Czechoslovakia.
Konyvkereskedes, Kilian, Budapest IV, Haris Koz 2, Hungary.
*Koschmann, A. H., Room 230, New Customhouse, Denver 2, Colorado.
Koucky, Frank, 5847 Cottage Grove, Chicago 37, Illinois.
*Kraus, Edward H., Mineralogical Laboratory, University of Michigan, Ann Arbor, Michigan.

Krishmamurthy, Esq., c/o Plaza Theatre, South Parade, Bangalore 1, India.
*Krishnan, Maharajapuram Sitaram, 27 Chowringhee, Calcutta, India.
*Krynine, Paul D., Pennsylvania State College, State College, Pennsylvania.
Kuenstler, Walter E., 410 Lafayette Avenue, Cliffside Park 8, New Jersey.
Kull, Srta. V., Perú 562, Buenos Aires, Argentina.
*Kulp, J. Laurence, Dept. of Geology, Columbia University, New York 27, New York.
Kunze, George, Dept. of Agronomy, Pennsylvania State College, State College, Pennsylvania.
Kurbatov, Joann D., Dept. of Physics, Ohio State University, Columbus, Ohio.
Kutina, Jan, Mineralogical Institute, Charles University, Albertov 6, Praha II, Czechoslovakia.

Lacy, Willard C., La Oroya, Peru.
Ladoo, Raymond B., 42 Huntington Road, Newton 58, Massachusetts.
*Landes, Kenneth K., Geology Department, University of Michigan, Ann Arbor, Michigan.
Lang, Andrew J., Jr., Dept. of Geology, University of Wichita, Wichita 6, Kansas.
Langford, Lawrence, 610 South Quivas Street, Denver 19, Colorado.
*Larsen, Esper S., Jr., Apt. 502-H, 3930 Connecticut Avenue, N.W., Washington 8, D. C.
*Larsen, Esper S., III, 7108 Radnor Road, Bethesda 14, Maryland.
Launder, William C., 54 Watson Road, Belmont 78, Massachusetts.
*Lausen, Carl, 504 East Fesler Street, Santa Maria, California.
Larkin, Robert G., 649 Bienveneda Avenue, Pacific Palisades, California.
Lawrence, Edmond F., P.O. Box 551, Torrance, California.
*Lee, Harley C., Basic Refractories, Inc., 845 Hanna Building, Cleveland 5, Ohio.
*Lee, O. Ivan, 2684 Boulevard, Jersey City 6, New Jersey.
Lee, Stephen B., 1801 Hood, Wichita 3, Kansas.
Leece, Arthur, 11400 Arden Avenue, Plymouth, Michigan.
Lefond, Stanley, 197 Prospect Avenue, Buffalo, New York.
Lehn, Joseph W., 6 Fort William Drive, Alexandria, Virginia.
Leith, Carlton J., 310 South Ward, Compton, California.
*Lemmon, Dwight M., U. S. Geological Survey, Washington 25, D. C.
Leonard, Benjamin F., U. S. Geological Survey, Box 7618, Lakewood Branch, Denver 15, Colorado.
Leonard, W. W., 589 Unadilla Street, Shreveport, Louisiana.
Letort, Yves, 3, Avenue Paul Doumer, Paris 16, France.
Levin, S. Benedict, 50 Crest Drive, Little Silver, New Jersey.
Lincoln, Earle R., Holberg, British Columbia, Canada.
*Lindberg, Mrs. Marie Louise, U. S. Geological Survey, Washington 25, D. C.
Lindhe, Henry E., P.O. Box #130, Austin 2, Texas.
Linville, P. E., 2017 Taft Avenue, Hollywood 28, California.
Livingston, John J., Quarters 32, Fort Belvoir, Virginia.
Loewenstein, Walter, Rua Bento Pareira, 326 Casa 1, Sao Paulo, Brazil, South America.
*Lonsdale, John T., Bureau of Economic Geology, University of Texas, Box B University Station, Austin 12, Texas.
*Lonsdale, Kathleen, University College, Gower Street, London W.C. 1, England.
*Lopez, Victor M., Apartado 3585, Caracas, Venezuela.
Lotz, Charles, 103 Grove Street, Meriden, Connecticut.

*Louderback, George D., Dept. of Mineralogy, University of California, Berkeley 4, California.
Loupekine, I. S., Dept. of Geology, The University, Bristol 8, England.
*Lovering, Thomas S., 8001 West 17th Avenue, Lakewood, Colorado.
Lowe, Kurt E., 196 Rockaway Parkway, Brooklyn 12, New York.
Lowe, W. Ross, Sudbury Assay Office, 256 Oak Street, Sudbury, Ontario, Canada.
Lucas, Elmer L., 219 West Eufala Street, Norman, Oklahoma.
*Lukesh, Joseph Stevens, 516 Riverside Avenue, Scotia, New York.
Lyden, Edward F. X., 51 East 702 Street, Shanks Village, Orangeburg, New York.
Lyons, John B., 7 Pleasant Street, West Lebanon, New Hampshire.

*Macdonald, Gordon A., U. S. Geological Survey, Hawaiian Volcano Observatory, Hawaii National Park, T. H.
Mackay, Ralph E., 2923 East Harrison Street, Seattle 2, Washington.
MacKenzie, David B., Geology Department, Princeton University, Princeton, New Jersey.
MacKenzie, G. S., Dept. of Geology, University of New Brunswick, Fredericton, N. B., Canada.
MacKenzie, William Scott, Geophysical Laboratory, 2801 Upton Street, N.W., Washington 8, D. C.
MacLachlen, J. C., Dept. of Geology, Princeton University, Princeton, New Jersey.
Magri, R. J., Jr., Corhart Refractories Company, 1600 W. Lee Street, Louisville 10, Kentucky.
Mandarino, Joseph A., 1034 North Keystone Avenue, Chicago 51, Illinois.
*Marble, John P., 3221 Macomb Street, N.W., Washington 8, D. C.
Marcin, Edward J., 10314 97th Avenue, Ozone Park, New York.
Marinos, George, Aminandrou 31 Koukaki, Athenes, Greece.
Markman, Harvey C., Curator of Geology, The Colorado Museum of Natural History, City Park, Denver 6, Colorado.
Markward, Ellen, 302 Beverly Apts., Columbia, Missouri.
*Marshall, C. Edmund, Dept. of Soils, College of Agriculture, University of Missouri, Columbia, Missouri.
Marshall, E. M., Redding Ridge, West Redding, Connecticut.
Marshall, Shadburn, U. S. Steel Company Laboratory, 234 Atwood Street, Pittsburgh, Pennsylvania.
*Martens, James H. C., Bureau of Mineral Research, Rutgers University, New Brunswick, New Jersey.
Martin, E. B., Westbury, Long Island, New York.
Martin, G. Louis, 104 West North Street, Sikeston, Missouri.
*Mason, Brian H., Dept. of Geology, Indiana University, Bloomington, Indiana.
Mason, Vincent I., 154 Beaumont Avenue, Newtonville 60, Massachusetts.
Mateer, W. D., Geology Department, Colorado School of Mines, Golden, Colorado.
Mather, Mrs. Bryant, P.O. Drawer 2131, Jackson, Mississippi.
Mathewson, Harris D., 357 "B" Obispo Street, Long Beach 14, California.
Matthews, Fred W., Canadian Industries, Ltd., Central Research Laboratory, McMasterville, Quebec, Canada.
Mattox, Richard B., Box 37, Amelia, Ohio.
Matzko, John J., 4208 Sheridan Street, Hyattsville, Maryland.
*Maugin, Charles, Laboratoire de Mineralogie de la Sorbonne, Paris 5, France.
Maurice, Charles S., 215 Cameron Avenue, Chapel Hill, North Carolina.

Mawby, M. A., Collins House, 360 Collins Street, Melbourne, Victoria, Australia.
*Maynard, James E., Dept. of Mineralogy, Syracuse University, Syracuse, New York.
McAndrew, John, Dept. of Geological Sciences, University of Toronto, Toronto 5, Canada.
*McCaughey, William J., Lord Hall, Ohio State University, Columbus, Ohio.
*McConnell, Duncan, Dept. of Mineralogy, Lord Hall, Ohio State University, Columbus 10, Ohio.
McCreery, G. L., Technical Director, Ohio Brass Company, Box 11, Barberton, Ohio.
McCreery, R. A., Agronomy Soils, State College of Washington, Pullman, Washington.
McIntosh, Alex O., Purvis Club, McMasterville, Quebec, Canada.
*McKinstry, Hugh E., Vanserg Laboratories, Cambridge 38, Massachusetts.
*McLaughlin, Donald H., 100 Bush Street, 26th Floor, San Francisco 4, California.
McLellan, Roy D., Research Department, American Smelting and Refining Company, Barber, New Jersey.
*McVay, Thomas N., School of Chemistry, University of Alabama, University, Alabama.
Meek, Ward Benjamin, 1610 Carr Street, Lakewood, Denver, Colorado.
*Meen, V. Ben, Royal Ontario Museum of Geology and Mineralogy, 100 Queen's Park, Toronto 5, Ontario, Canada.
Meixner, Heinz, Bergdirektion Hüttenberg, Post Knappenberg, Carinthia, Austria.
Mellis, Otto, Stockholms Hogskolas Mineralogiska Institute, Kungstengatan 45, Stockholm, Sweden.
Mencher, Ely, c/o Socony-Vacuum Oil Company of Venezuela, Apartado 246, Caracas, Venezuela.
Mendoza, O. Baca, Box 286, Cuzco, Peru.
Mergner, John L., 5519 Nevada Avenue, N.W., Washington, D. C.
*Merritt, Clifford A., University of Oklahoma, Norman, Oklahoma.
Merritt, Phillip L., 450 Clinton Avenue, Brooklyn 5, New York.
*Mertie, John B., Jr., U. S. Geological Survey, Room 12, Building 213, Naval Gun Factory, Washington, D. C.
*Merwin, Herbert E., Geophysical Laboratory, 2801 Upton Street, N.W., Washington 8, D. C.
Meyer, Charles, Box 597, Butte, Montana.
Meyers, T. R., Dept. of Geology, University of New Hampshire, Durham, New Hampshire.
Michell Bawden, F. C., c/o Geology Department, New Consolidated Goldfields, Box 1167, Johannesburg, South Africa.
*Michener, Charles E., International Nickel Company of Canada, Ltd., Copper Cliff, Ontario, Canada.
Michenfelder, Frank, c/o Sun Oil Company, Beaumont, Texas.
Midgley, H. G., Building Research Station, Garston, Watford, Herts, England.
*Mielenz, Richard C., Bureau of Reclamation, Customhouse, Denver 2, Colorado.
Mikami, Harry M., E. J. Lavino & Company, Box 29, Norristown, Pennsylvania.
Miller, Clarence E., Dept. of Geology & Geography, University of Rhode Island, Kingston, Rhode Island.
Miller, F. Stuart, 49 Mendota Avenue, Rye, New York.
Miller, Roswell III, 9 Campbelton Circle, Princeton, New Jersey.
*Miller, William J., 436 West Willow Street, Stockton 19, California.
*Milton, Charles, U. S. Geological Survey, Washington 25, D. C.
*Miser, Hugh D., U. S. Geological Survey, Washington 25, D. C.
*Moneymaker, Berlen C., c/o T. V. A. Geologic Division, 620 Union Building, Knoxville, Tennessee.

*Montgomery, Arthur, 277 Park Avenue, New York 17, New York.
*Moore, Elwood S., University of Toronto, Toronto 5, Canada.
Moore, Henry J., III, 1354 E. South Temple Street, Salt Lake City 2, Utah.
Moore, John J., 133 Blue Hills Parkway, Milton, Massachusetts.
Moore, Kenneth P., 533 South Grant, Casper, Wyoming.
Morgan, Leonard A., 217 South Atlantic Avenue, Haddonfield, New Jersey.
Morley, Russell A., 399 North 18 Street, Salem, Oregon.
*Morris, Frederick K., Arctic, Desert, Tropic Inf. Center, Air University Library, Maxwell AFB, Alabama.
Morrison, Bert C., 409 Orchard, Bayard, New Mexico.
Mortola, Dra. Edelmire, Junin 1367, Piso 3, A, Buenos Aires, Argentina.
Moyd, Louis, Minerals, Ltd., Bancroft, Ontario, Canada.
Mrose, Mary E., 846 Highland Avenue, Medford, Massachusetts.
Mullner, Henry C., 141 Ackerman Avenue, Clifton, New Jersey.
*Murata, K. J., U. S. Geological Survey, Washington 25, D. C.
*Murdoch, Joseph, University of California at Los Angeles, Los Angeles 24, California.
Murowchick, Bernard L., 637 Sagamore Street, Lakeland, Florida.
Murphy, Richard, Newmont Mining Corporation of Canada, Room 1721, 5 King Street West, Toronto, Canada.
Murray, Albert N., Dept. of Geology, University of Tulsa, Tulsa 4, Oklahoma.
Murray, Haydn H., Dept. of Geology, Indiana University, Bloomington, Indiana.
Mutch, A. D., 363 Ramsay Road, Sudbury, Ontario, Canada.
Meyers, Alfred T., U. S. Geological Survey, Denver Federal Center, Denver 14, Colorado.

*Nagelschmidt, Gunter, Birchtrees, Ninelands, Hathersage near Sheffield, England.
Naidu, P. R. J., Dept. of Geology, Central College, Bangalore, India.
Nair, K. V. Krishnan, Division of Mineral Survey & Research, University of Travancore, Trivandrum, India.
Nalle, Peter B., 4443 Central Avenue, Riverside, California.
Neiman, Robert, 505 E. Chestnut Street, Louisville 2, Kentucky.
*Nel, Hans Jurie, Dept. of Geology, University of Pretoria, Pretoria, South Africa.
Nelson, Harry E., 1020 West Silver Street, Butte, Montana.
Neuerburg, George J., 12615 Chandler Boulevard, North Hollywood, California.
*Newhouse, Walter H., Dept. of Geology, University of Chicago, Chicago, Illinois.
Newman, George H., Marsman & Company, P.O. Box 297, Manila, Philippine Islands.
Nieset, Carl F., St. Joseph College, Collegeville, Indiana.
Nisson, William H., 815 D Street, Petaluma, California.
*Noble, James A., California Institute of Technology, Pasadena 4, California.
*Nolan, Thomas B., U. S. Geological Survey, Washington 25, D. C.
Normart, Joseph J., 438 South 3rd Avenue, Tucson, Arizona.
Northup, Allen, 10 Godet Place, Morristown, New Jersey.
Norton, James J., Mineral Deposits Branch, U. S. Geological Survey, Washington 25, D. C.
*Nowacki, W., H. Sahlistr. 6, Min. Inst. Univ., Bern, Switzerland.
*Nuffield, Edward W., Dept. of Geological Sciences, University of Toronto, Toronto 5. Canada.
Nygreen, Paul W., 5643 Brooklyn Avenue, Seattle 5, Washington.

Odom, W. R., Star Route A, Box 32-0, Austin, Texas.
Oke, William C., 9115 South Harvard Boulevard, Los Angeles 47, California.

O'Leary, W. J., 300 East 25 Street, Wilmington, Delaware.
Olson, J. C., U. S. Geological Survey, Denver Federal Center, Denver, Colorado.
OMori, Kei-Ichi, Institute of Mineralogy, Petrology and Economic Geology, Tohoku University, Katahiracho, Sendai, Japan.
Onay, Togan S., Iller Kooperatififi, Birinci Sokak 31, Akara, Turkey.
Ootsubo, Yoshio, 10-22, Nishikata-machi, Bunkyo-ku, Tokyo, Japan.
*Orcel, Jean, Museum d'Histoire Naturelle, 61 Rue de Buffon, Paris V, France.
Ortega, Francisco Barroso, Lab. No. 2 de Mineralogie, Facultad de Ciencias, Universidad de la Habana, La Habana, Cuba.
Osberg, Philip H., Division of Geology, Pennsylvania State College, State College, Pennsylvania.
*Osborn, Elburt Franklin, Dept. of Earth Sciences, School of Mineral Industries, Pennsylvania State College, State College, Pennsylvania.
Ottsen, Laurine, Filderstr. 82, Moers, Niederrh., Germany.
Over, Edwin, c/o R.L. Chadbourne, 320 East Uintah, Colorado Springs, Colorado.

*Pabst, Adolf, Dept. of Geology, University of California, Berkeley 4, California.
Paclt, J., Lamacaka cesta 5, Bratislava 9, Czechoslovakia.
Page, James J., RFD 2, Pike, New Hampshire.
*Page, Lincoln R., 2217 Brentwood Street, Lakewood, Denver 15, Colorado.
*Palache, Charles, 106 Appleton Street, Cambridge 38, Massachusetts.
*Park, Charles Frederick, Jr., Dept. of Geology, Stanford University, California.
*Parker, Robert L., Technische Hochschule, Sonneggstrasse 5, Zürich 6, Switzerland.
Parnau, John L., 1342 North Lincoln Street, Stockton, California.
*Parrish, William, Philips Laboratories, Inc., Irvington-on-Hudson, New York.
*Parsons, Arthur L., Royal Ontario Museum of Mineralogy, 100 Queen's Park, Toronto 5, Ontario, Canada.
*Parsons, Willard Hall, Dept. of Geology, Wayne University, Detroit 1, Michigan.
Pask, Joseph A., Division of Mineral Technology, University of California, Berkeley 4, California.
*Patterson, A. L., Institute for Cancer Research, Fox Chase, Philadelphia 11, Pennsylvania.
Patterson, C. Meade, 382 Woodland Avenue, Oakmont, Pennsylvania.
Pearce, D. W., 1814 Hunt, Richland, Washington.
Pearl, Richard M., Colorado College, Colorado Springs, Colorado.
Peckham, Bob, 132 Bartlett Avenue, Woodland, California.
*Pecora, William T., U. S. Geological Survey, Washington 25, D. C.
*Pegau, Arthur A., 1808 Winston Avenue, Charlottesville, Virginia.
Pelto, Chester R., MRD Laboratory, Corps of Engineers, 18th & Howard Streets, Omaha 2, Nebraska.
Pendleton, Norman H., 11 Pryce Street, Santa Cruz, California.
*Peoples, Joe Webb, Dept. of Geology, Wesleyan University, Middletown, Connecticut.
*Pepinsky, Raymond, Research Professor of Physics, Pennsylvania State College, State College, Pennsylvania.
Periera, Moacir D., Rua Theofilo Otoni, 15 7and, s/712, Rio de Janeiro, Brazil.
Perloff, Louis, 1831 Nissen Building, Winston-Salem, North Carolina.
Perry, Elwyn L., Dept. of Geology, Williams College, Williamstown, Massachusetts.
Perusek, Cyril J., 24-5 W. Cuthbert, Midland, Texas.
Peterson, Nels P., Box 1211, Globe, Arizona.
Petretic, George J., 212 Penfield Place, Dunellen, New Jersey.
Phair, George, 2230 Savannah Terrace, S.E., Apt. 33, Washington 20, D. C.

*Phemister, T. C., Dept. of Geology and Mineralogy, University of Aberdeen, Marischal College, Aberdeen, Scotland.
Philbrook, Mrs. P. M., P.O. Box 184, Sacramento 1, California.
Phoenix, Robert, 103 Sharpe Avenue, Toronto 13, Canada.
Pinson, William H., Jr., 252 Westgate West, Cambridge 39, Massachusetts.
Pitha, Carl A., Dept. of Chemistry, Northwestern University, Evanston, Illinois.
Pizarro, Carlos Jiles, Casilla 23, Ocalle, Chile, South America.
Platt, Philip L., 74 Beacon Hill Road, Port Washington, New York.
Plummer, Wayne L., 607 East 23 Street, Hibbing, Minnesota.
Pocovi, Antonio, Rivadavia 3567, Santa Fe, Argentina.
Poestkoke, J. Fred, 926 East 2, Casper, Wyoming.
Poire, Inna V., U. S. Geological Survey, FWA Building, Room 1228, Washington 25, D. C.
*Poldervaart, Arie, Geological Survey, Lobatsi, Bechuanaland Protectorate, S. A.
Poole, S. W., 1346 22 Street N.W., Canton 3, Ohio.
Poole, W. E., 127 Alabama Street, Spartanburg, South Carolina.
Porter, Percy Waterman, Box 122, Cave Creek, Arizona.
*Postel, A. Williams, 35 Farragut Avenue, Kensington, Maryland.
*Pough, Frederick H., American Museum of Natural History, 77th Street and Central Park West, New York 24, New York.
Prather, Robert W., Dept. of Physics, Oregon State College, Corvallis, Oregon.
Presgrave, Cyril, Gwynedd Valley, Pennsylvania.
Price, Walter E., Jr., 7388 Goodwood Avenue, Baton Rouge, Louisiana.
*Prider, Rex T., Dept. of Geology, University of Western Australia, Nedlands, Western Australia.
Prien, Edwin L., 1101 Beacon Street, Brookline 46, Massachusetts.
Prucha, John James, 17 Warren Street, McKnownville, Albany 3, New York.
Puig, Joseph A., 7214 Sixth Avenue, Brooklyn 9, New York.

*Quensel, Percy D., Stockholms Högskola, Stockholm, Sweden.
Quest, H. J. M., Box 54, Klerksdorp, Transvaal, South Africa.
*Quinn, Alonzo W., Dept. of Geology, Brown University, Providence, Rhode Island.

*Rabbitt, John C., U. S. Geological Survey, Washington 25, D. C.
Rahdo, Hannes, Box 205, Vader, Washington.
Ramberg, Hans, Dept. of Geology, University of Chicago, Chicago 37, Illinois.
*Ramdohr, Paul, Mineralogisch-Petrographisches Institut der Universität, Hauptstrasse 47–51, Heidelberg, Germany.
*Ramsdell, Lewis S., Mineralogical Laboratory, University of Michigan, Ann Arbor, Michigan.
*Rankama, Kalervo, Institute of Geology, University of Helsinki, Snellmanink 5, Helsinki, Finland.
Rath, A. Donald, 25 Windsor Road, Brookline 46, Massachusetts.
Redden, Jack A., Box 71, Custer, South Dakota.
Reed, Frank S., 3118 Rittenhouse Street, N.W., Washington, D. C.
Reeves, Augustus, Clayton, Ohio.
*Reinhard, Max, Mineralogical Institute, University of Basel, Basel, Switzerland.
Remick, Jerome H., Newman House, 1301 Ruby Avenue, Houghton, Michigan.
Reyes, Jorge Munoz, Apartado 267, La Paz, Bolivia.
Rice, H. M., Chemistry Division, Central Experimental Farm, Ottawa, Canada.
Richmond, James F., 742 Forest Avenue, Palo Alto, California.

*Richmond, Wallace E., Jr., RD 2, Hillsboro, New Hampshire.
Ridge, John D., Lemont, Centre County, Pennsylvania.
Rigsby, George P., Division of Geological Sciences, California Institute of Technology, Pasadena 4, California.
Rinn, Harold W., 2917 Braley Court, Midland, Michigan.
Ritchie, Everett J., c/o Eagle-Picher Company, Research Laboratories, Joplin, Missouri.
Roberts, Carroll Norton, Dept. of Geology, Indiana University, Bloomington, Indiana.
Roberts, Fred, Jr., 1106 West Newmark, Monterey Park, California.
Roberts, W. L., 628 Harter Drive, Rapid City, South Dakota.
Robertson, E. C., c/o Dept. of Geology, Harvard University, Cambridge 38, Massachusetts.
Robertson, Forbes, Montana School of Mines, Butte, Montana.
Robinson, G. D., Section of General Geology, U. S. Geological Survey, Denver Federal Center, Denver 14, Colorado.
Rode, K. P., University Department of Geology, M B College, Udepur, India.
Rodekohr, W. J., 25 Elgin Street, Alhambra, California.
Roedder, Edwin W., Dept. of Mineralogy, University of Utah, Salt Lake City, Utah.
*Rogers, Austin P., 2412 Durant Avenue, Apt. 109, Berkeley, California.
Rollins, Ross, 79 Cypress Street, Brookline, Massachusetts.
Rolshausen, F. W., P.O. Box 2180, Houston 1, Texas.
Rooney, John T., Box 358, Buffalo, New York.
Rose, Robert L., 1425 Dutton Avenue, Santa Rosa, California.
Rosenfeld, John L., 1014 Morrell Avenue, Rolla, Missouri.
*Rosenholtz, Joseph L., Rensselaer Polytechnic Institute, Troy, New York.
Rosenkrans, R. R., 373 Albert, Shreveport, Louisiana.
Rosevear, F. B., Chemical Division, The Procter & Gamble Company, Cincinnati 17, Ohio.
*Ross, Clarence S., U. S. Geological Survey, Washington 25, D. C.
Ross, Mrs. Kip, Broadmoor #305, 3601 Connecticut Avenue, N.W., Washington 8, D. C.
Rossi, Juan Jose, Rodriguez Pena 1043, Buenos Aires, Argentina.
Roth, Robert S., 1630 Park Road, N.W., Washington, D. C.
*Rowland, Richards A., Shell Oil Company, P.O. Box 2099, Houston 1, Texas.
Rowley, Elmer B., 214 Ridge Street, Glens Falls, New York.
Roy, Chalmer J., Dept. of Geology, Iowa State College, Ames, Iowa.
Royal, H. F., Champion Spark Plug Company, 8525 Butler Avenue, Detroit 11, Michigan.
Royer, Louis, Laboratoire de Min. et Crist. de l'Université, Algiers, Algeria.
Runner, Delmar G., 4606 47th Street, N.W., Washington 16, D. C.
*Runner, Joseph J., Geology Building, University of Iowa, Iowa City, Iowa.
*Russell, Arthur E., Swallowfield Park, Reading, Berkshire, England.
Russell, Robert T., U. S. Bureau of Reclamation, 460 N. High Street, Salem, Oregon.
*Rutherford, Ralph L., Dept. of Geology, University of Alberta, Edmonton, Alberta, Canada.

Sachs, Walter P., 254 Bloomfield, Avenue, Caldwell, New Jersey.
Safford, Arthur T., Jr., 328 North Steele Road, West Hartford, Connecticut.
*Sahama, Thure Georg, Institute of Geology, University of Helsinki, Snellmanink 5, Helsinki, Finland.

Sahlin, Bergsing. Carl-Ingemar, Villastigen 1, Kiruna, Sweden.
Saletel, Louis, University of Dayton, Dayton 9, Ohio.
Sambonsugi, Miyoji, 84 Hamadacho, Fukushima City, Japan.
*Sampson, Edward, Guyot Hall, Princeton University, Princeton, New Jersey.
Sampter, E. L., 49 West 45th Street, New York 19, New York.
*Sandell, Ernest B., School of Chemistry, University of Minnesota, Minneapolis 14, Minnesota.
San Martín y Sáenz, René, Laboratorio de Mineralogia No. 1, Facultad de Ciencias, Universidad de la Habana, La Habana, Cuba.
Saunders, Harold A., Hotel Heppner, Heppner, Oregon.
Sawyer, Harold H., 514 W. 12th Street, Anderson, Indiana.
Saylor, Pauline A., 20144 East Arrow Highway, Covina, California.
Schaa, Ferdinand A., Jr., Saxon Avenue, Bay Shore, New York.
*Schairer, John F., Geophysical Laboratory, 2801 Upton Street, N.W., Washington 8, D. C.
*Schaller, Waldemar T., U. S. Geological Survey, Washington 25, D. C.
Scheid, Vernon E., Mackay School of Mines, University of Nevada, Reno, Nevada.
Schermeister, Leo, University of Illinois College of Pharmacy, 808 S. Wood Street, Chicago, Illinois.
Schlecht, William G., U. S. Geological Survey, Washington 25, D. C.
Schlocker, J., U. S. Geological Survey, 105 Old Mint Building, San Francisco, California.
*Schmitt, Harrison A., Cottage Sanatorium Road, Silver City, New Mexico.
Schmitter, V. Eduardo, Anaxagoras 921, Mexico 12, D.F., Mexico.
*Schneider, Philip E., 46 Blue Hills Avenue, Hartford 5, Connecticut.
Schoenlaub, R. A., Sylvester and Company, Terminal Tower, Cleveland 13, Ohio.
*Schoep, Alfred, 97 Baudeloo Street, Ghent, Belgium.
Schortmann, Raymond E., 306 Main Street, Easthampton, Massachusetts.
Schwartz, B., 1884 Monroe Avenue, Bronx, New York 57, New York.
*Schwartz, George M., Dept. of Geology, University of Minnesota, Minneapolis 14, Minnesota.
Scotford, David M., Geology Department, Miami University, Oxford, Ohio.
Scott, Donald W., Continental Sales & Equipment Company, Box 438, Hibbing, Minnesota.
Scott, Glenn R., U. S. Geological Survey, En. Section, Denver Federal Center, Denver 14, Colorado.
Scott, Horace A., 2208 N. Ross, Santa Ana, California.
Seager, A. F., 94 Ranelagh Road, Ealing, London WF, England.
Seaman, David Martin, 51 Ralph Mann Drive, Stoughton, Massachusetts.
Segeler, Curt G., 284 East 16th Street, Brooklyn 26, New York.
*Seifert, H., Mineralogical-Petrographic Institute, Westphalian Land University (21a), Münster i.W., Germany.
*Shand, S. James, 1 Fintry Place, Broughty-Ferry, Dundee, Scotland.
Sharp, Kenneth R., S.M.H.E.A., Cooma 4S, New South Wales, Australia.
*Shaub, Benjamin M., 159 Elm Street, Northampton, Massachusetts.
Sheheen, Alexander T., 80 Elm Street, Hornell, New York.
Sheridan, Douglas M., U. S. Geological Survey, Box 7618, Lakewood Branch, Denver 15, Colorado.
Sherman, Walter C., 27 Doro Place, Rumford 16, Rhode Island.
Shimamoto, Mrs. K., 1330 Congress Street, S.E., Washington 20, D. C.

Shipley, Robert M., Gemological Institute of America, 541 S. Alexandria Avenue, Los Angeles 5, California.
Shipton, W. D., Dept. of Geology, Washington University, St. Louis 5, Missouri.
*Short, Max N., Dept. of Geology, University of Arizona, Tucson, Arizona.
Shortle, Walter C., 65 Lincoln Street, Laconia, New Hampshire.
Shurtz, Robert F., Box 788, Henderson, Nevada.
Sidwell, Raymond, Dept. of Geology, Texas Technical College, Lubbock, Texas.
Siegrist, Marie, 1431 Iris Street, N.W., Washington 12, D. C.
Sievers, D. C., 1626 Woodside Avenue, Kingsport, Tennessee.
Silberstein, M. L., 1607 Francis Street, Houston 4, Texas.
Silver, Leon T., Division of Geological Sciences, California Institute of Technology, Pasadena 4, California.
Silverman, Mrs. Jeanne, 1224 High Street, Whittier, California.
Simcock, J. H., Cranford, Lovatt Avenue, Milehouse, Newcastle, Staffordshire, England.
Simmons, Edward E., Jr., 455 S. Oakland Avenue, Pasadena 5, California.
Simons, Frank S., U. S. Geological Survey, Room 4209, GSA Building, Washington 25, D. C.
*Singewald, Quentin D., U. S. Geological Survey, Washington 25, D. C.
Skaritza, Joseph, Box 157, Great Bend, Pennsylvania.
Skea, E. M., P.O. Box 46, Pilgrims Rest, Transvaal, South Africa.
*Slawson, Chester B., Mineralogical Laboratory, University of Michigan, Ann Arbor, Michigan.
Smedes, Harry W., Dept. of Geology, Kansas State College, Manhattan, Kansas.
Smith, Alan R., Geology Department, International Nickel Company, Copper Cliff, Ontario, Canada.
Smith, D. T., Rensselaer Polytechnic Institute, Troy, New York.
Smith, Edward S. C., Geology Laboratory, Union College, Schenectady 8, New York.
Smith, O. C., 5157 Santa Ana Street, Bell, California.
Smith, Robert L , 4756 F. O. Boulevard, Fair Oaks, California.
*Smith, W. S. Tangier, 1231 Hoover Street, Menlo Park, California.
*Smith, Ward C., 99 El Camino Real, Berkeley 5, California.
Smythe, Frank W., Jr., 4065 Grandview Boulevard, Memphis, Tennessee.
*Snelgrove, Alfred K., Dept. of Geology, Michigan College of Mining and Technology, Houghton, Michigan.
Snow, Roland B., Research Laboratory, U. S. Steel Corporation, Kearny, New Jersey.
Socolow, Arthur A., Geology Department, Boston University, 725 Commonwealth Avenue, Boston, Massachusetts.
*Spence, Hugh S., 222 Argyle Avenue, Ottawa, Ontario, Canada.
Sperry, Arthur B., Dept. of Geology, Kansas State College of Agriculture and Applied Science, Manhattan, Kansas.
Spiller, R. C., Dept. of Mineralogy, University Museum, Oxford, England.
Spiroff, Kiril, Houghton, Michigan.
Spotts, John H., Senath, Missouri.
Springer, W. M., Bristol-Myers Company, 225 Long Avenue, Hillside, New Jersey.
Sproat, Ira E., 58 Washington Avenue, Westport, Connecticut.
Staatz, Mortimer H., Box 7618, Lakewood Branch, Denver 15, Colorado.
Stadnichenko, Taisia, U. S. Geological Survey, Washington 25, D. C.
*Stanton, Gilman S., 245 West 104 Street, New York 25, New York.
Stanwood, Creighton B., 12 Kilburn Road, Belmont 78, Massachusetts.
*Staples, Lloys W., Dept. of Geology, University of Oregon, Eugene, Oregon.

*Stark, John T., Dept. of Geology, Northwestern University, Evanston, Illinois.
Stead, Frank, U. S. Geological Survey, Washington 25, D. C.
Steedman, R. D., 17 Saville Row, Newcastle on Tyne, England.
Steel, George E., 37 Mason Boulevard, Toronto, Ontario, Canada.
Steen, Charles A., 3803 Arbor Street, Houston 4, Texas.
Stephano, C. S., 1016 Walnut Street, Philadelphia 7, Pennsylvania.
Stern, Thomas W., U. S. Geological Survey, Geochemistry & Petrology, Washington 25, D. C.
Stevens, D. H., 2557 Casitas Avenue, Altadena, California.
*Stevens, Rollin E., U. S. Geological Survey, Washington 25, D. C.
*Stevenson, John S. Dept. of Geological Sciences, McGill University, Montreal, Canada.
Stewart, David B., 401 William James Hall, Cambridge 38, Massachusetts.
*Stewart, Duncan, Dept. of Geology, Carleton College, Northfield, Minnesota.
Stewart, Glenn W., Dept. of Geology, University of New Hampshire, Durham, New Hampshire.
Stieff, Lorin R., 2242 Washington Avenue, Apt. 102, Silver Spring, Maryland.
Stilwell, Stephen N., 315 Empire Building, Knoxville, Tennessee.
Stivers, Glen H., Box 581, Tustin, California.
Stobbe, Helen, Norris House, Smith College, Northampton, Massachusetts.
Stockman, A. J., Ruby, Alaska.
*Stoiber, Richard E., McKenna Road, Norwich, Vermont.
Stone, Robert L., Dept. of Ceramic Engineering, University of Texas, Austin 12, Texas.
*Stow, Marcellus H., 405 Massie Street, Lexington, Virginia.
*Stringham, Bronson F., University of Utah, Salt Lake City, Utah.
*Strock, Lester W., 21 Madison Avenue, Saratoga Springs, New York.
*Strunz, Hugo, Mineralogisch-Geologisches Institut, Regensburg, Germany.
Stucker, P., Riectalstrasse 3, Zofingen, Switzerland.
*Stuckey, Jasper L., 1911 Sunset Drive, Raleigh, North Carolina.
Stugard, Frederick, Jr., 460 Hoyt Street, Lakewood, Colorado.
Subramaniam, A. P., Dept. of Geology, Princeton University, Princeton, New Jersey.
Sudo, Toshio, Geological Institute, Faculty of Science, Tokyo University, Tokyo, Japan.
Sun, Ming-Shan, Bureau of Mines and Mineral Resources, Campus Station, Socorro, New Mexico.
Swinnerton, A. C., 145 West Limestone Street, Yellow Springs, Ohio.
Swinney, C. M., School of Mineral Sciences, Stanford University, California.
*Switzer, George, U. S. National Museum, Washington 25, D. C.
Szadeczky-Kardoss, Elmer, Inst. Mineralogy & Geology, Sopron University, Sopron, Hungary.

*Taber, Stephen, 1518 Deans Lane, Columbia 5, South Carolina.
Takatsui, Fumio, 14521 27 N.E., Seattle 55, Washington.
Tamura, Tsumeo, Soils Dept., University of Wisconsin, Madison, Wisconsin.
Tatge, Eleanor, McLean, Virginia.
Tauber, J. A., P.O. Box 29, Norristown, Pennsylvania.
*Tavora, Elysiario, Faculdade Nacional de Filosofia, Universidade do Brasil, Av. Pres. Antonio Carlos 40, Rio de Janeiro, Brazil.
Taylor, Albert J., 1204 West 44th Street, Austin 5, Texas.
Taylor, Allen O., 756 East 2d South, Apt. 26, Salt Lake City, Utah.
Taylor, Edward D., HQ, NWHS, Whitehorse, Yukon Territory, Canada.
Taylor, James A., 38 Douglas Road, Glen Ridge, New Jersey.

Taylor, Ralph E., Box 2180, Houston 1, Texas.
*Taylor, W. H., The Cavendish Laboratory, Crystallographic Section, Free School Lane, Cambridge, England.
*Thibault, N. W., Norton Company, Worcester, Massachusetts.
Thomas, W. Hersey, 145 East Gorgas Lane, Mt. Airy, Philadelphia, Pennsylvania.
Thompson, Clyde L., Hays Laboratory, Pittsburgh 7, Pennsylvania.
Thompson, James B., Dept. of Mineralogy, Harvard University, Cambridge 38, Massachusetts.
Thompson, Mary E., U. S. Geological Survey, Naval Gun Factory, Washington 25, D. C.
Thompson, Robert, Box 913, Cobalt, Ontario, Canada.
*Thompson, Robert M., Dept. of Geology & Geography, University of British Columbia, Vancouver, B. C., Canada.
Thurston, William R., Compania Minera de Penoles, S. A., Apartado 251, Monterrey, N.L., Mexico.
Titcomb, Jane, U. S. Geological Survey, Naval Gun Factory, Washington 25, D. C.
*Tolman, Carl, Dept. of Geology, Washington University, St. Louis 5, Missouri.
Toman, Larry, Jr., Box 452-C, M.R., Red Bank, New Jersey.
Tomasino, Peter, 3130 West Mitchell, Milwaukee 15, Wisconsin.
Tomlinson, W. Harold, 260 North Rolling Road, Springfield, Pennsylvania.
Tooker, Edwin W., 234 Natural History Building, University of Illinois, Urbana, Illinois.
*Toothaker, Charles R., Commercial Museum, 24th and Spruce Streets, Philadelphia 4, Pennsylvania.
Trainer, David W., Jr., 48 Payne Street, Hamilton, New York.
Trainer, J. N., Brewster, New York.
Trautz, Otto R., Dept. of Chemistry, New York University College of Dentistry, 209 East 23d Street, New York 10, New York.
Travis, Russell B., International Petroleum Company, Ltd., Talara, Peru.
Trefethen, Joseph M., New Engineering Building, University of Maine, Orono, Maine.
Tremblay, Abbe J. A., Seminaire de Chicoutimi, Comte de Chicoutimi, P. Q., Canada
Trimble, Clinton E., 242 Heliotrope Avenue, Corona Del Mar, California.
Trudell, Harry W., 1309 Highland Avenue, Abington, Pennsylvania.
Trumpour, Harry J., 528 South 14 Street, Corvallis, Oregon.
Trites, Albert F., Jr., 505 Arapahoe Street, Golden, Colorado.
*Tunell, George, 801 Toyopa Drive, Pacific Palisades, California.
*Turneaure, F. S., 1905 E. Stadium Drive, Ann Arbor, Michigan.
*Turner, Francis J., Bacon Hall, University of California, Berkeley 4, California.
Turobinski, T. J., 1965 East 83 Street, Cleveland, Ohio.
Tuthill, Isabel H., Rocky Point, Long Island, New York.
Tuttle, Orville F., Geophysical Laboratory, 2801 Upton Street, Washington 8, D. C.
Twenhofel, William S., U. S. Geological Survey, Box 2659, Juneau, Alaska.
Tweto, Ogden, U. S. Geological Survey, 200 New Customhouse, Denver 2, Colorado.

Usi, Prof., Kyoto Kogei-Seni-Daigaku-Kogeigakubu, Matsugasaki, Kyoto, Japan.

Valentine, Grant M., 1310 Rockefeller Avenue, Everett, Washington.
Valentine, Wilbur G., Dept. of Geology, Brooklyn College, Brooklyn, New York.
Vallance, Thomas G., 57 Auburn Street, Sutherland, New South Wales, Australia.
Vance, Louis W., 1002 Palm Avenue, South Pasadena, California.
Van Cott, H. Corbin, 185 Fulton Street, Corning, New York.

Vandereike, Paul, Rt. 2, Box 1168, Vista, California.
Van der Leur, Rene, 341 East Brewer Street, East Hartford 8, Connecticut.
Van Horn, Earl C., Box 221, Murphy, North Carolina.
Van Horn, Maurice H., Research Laboratories, Eastman Kodak Company, Kodak Park, Rochester 4, New York.
Van Vlack, Lawrence, 27 McGarity Road, Park Forest, Chicago Heights, Illinois.
Vaughan, Allan C., U. S. Smelting & Refining Company, Midvale, Utah.
Venuto, Joseph, 1421 Sterling Place, Brooklyn 13, New York.
Vesignie, Jean Paul Louis, 22 Rue du General Foy, Paris VIII, France.
Vhay, John Stewart, S. 157 Howard, Spokane, Washington.
Vickers, A. Eric J., Wynnstay, Millfield Road, Whickham, Newcastle-Upon-Tyne, England.
Vietzke, Fred W. C., RFD #1, Box 10, Rapid River, Michigan.
Vishweswar, M., Post Box no. 714, Bombay 1, India.
*Vitaliano, Charles J., Dept. of Geology, Indiana University, Bloomington, Indiana.
Vogt, Arno R., Box 348, New London, Connecticut.
Volesky, Gustav, Jr., 38 Narodni, Prague 2, Czechoslovakia.
von Vultee, Joachim, Schlosstrasse 17, Berlin-Steglitz, Germany.
Vreeland, Frederick K., 36 Magee Avenue, Mill Valley, California.

Wade, F. Alton, Dept. of Geology, Miami University, Oxford, Ohio.
*Wager, Lawrence R., Dept. of Geology and Mineralogy, University Museum, Oxford, England.
*Wahlstrom, Ernest E., Dept. of Geology, University of Colorado, Boulder, Colorado.
Waldo, Allen W., 1335 North Van Buren, Stockton 19, California.
*Waldschmidt, William A., 1901 W. College, Midland, Texas.
Wall, Milford W., 12 Euston Street, Brookline, Massachusetts.
Wallaca, Robert E., Dept. of Geology, State College of Washington, Pullman, Washington.
*Wallace, Robert C., Queen's University, Kingston, Ontario, Canada.
Wandke, Alfred D., Box 1512, Prescott, Arizona.
Warner, Robert E., 2980 Pingree Avenue, Detroit, Michigan.
Warrach, Henry C., P.O. Box 162, Hammond, Indiana.
Warshaw, Mrs. Charlotte M., Division of Geophysics and Geochemistry, Dept. of Earth Sciences, State College, Pensylvania.
Washken, Edward, 2076 Massachusetts Avenue, Cambridge 40, Massachusetts.
*Wasserstein, Benno, Geological Survey, Union of South Africa, Pretoria, South Africa.
*Watanabe, Takeo, Tokyo Imperial University, Tokyo, Japan.
*Waters, Aaron C., Box 1738, Stanford University, California.
Waterston, Thomas F., 435 Metcalfe Avenue, Westmount, Quebec, Canada.
*Watson, Edward H., Dept. of Geology, Bryn Mawr College, Bryn Mawr, Pennsylvania.
*Watson, Kenneth DeP., Dept. of Geology, University of California, Los Angeles 24, California.
*Wayland, Russell G., Combined Coal Control Group, US-HICOG, Box 590, APO 80, c/o Postmaster, New York, New York.
*Webb, Robert W., Dept. of Physical Science, University of California, Santa Barbara, California.
Weeks, Mrs. Alice D., Geochemistry & Petrology, U. S. Geological Survey, Washington 25, D. C.

Weeks, Charles A., Red Gate Lane, Meredith, New Hampshire.
Weidhaas, Ernest, 255 Corlies Avenue, Pelham 65, New York.
Weis, Paul L., School of Geology, University of Virginia, Charlottesville, Virginia.
Weiss, E. Joseph, Dept. of Ceramic Engineering, University of Texas, Austin 12, Texas.
Weissenborn, A. E., So. 157 Howard, Spokane 8, Washington.
Weitz, John H., Dept. of Geology, Lehigh University, Bethelehem, Pennsylvania.
Wellnitz, Anna M., Brooklyn College, Bedford & H Avenues, Brooklyn 10, New York.
Wells, Ralph G., 379 Woodcliff Circle, Pittsburgh 16, Pennsylvania.
Welt, Ruth M., 132 Bay 31st Street, Brooklyn 14, New York.
Wenden, Henry E., 16 Normandy Road, Woodhaven, Lexington 73, Massachusetts.
*West, Cutler D., Polaroid Corporation, 718 Main Street, Cambridge 38, Massachusetts.
*Wherry, Edgar T., 137 Maplewood Avenue, Philadelphia 44, Pennsylvania.
Whitaker, George C., 825 East Schaaf Road, RD 3, Brooklyn Station, Cleveland, Ohio.
White, Charles E., Dept. of Chemistry, University of Maryland, College Park, Maryland.
White, James C. M., 203 Carnegie Place, Pittsburgh 8, Pennsylvania.
White, J. H., 938 Northampton Drive, Silver Spring, Maryland.
White, W. Arthur, Illinois Geological Survey, Urbana, Illinois.
White, W. E., 552 Booth Street, Ottawa, Canada.
Whitmore, D. R. E., C.M.S. Company, Box 1340, Prince Rupert, British Columbia, Canada.
Whittard, W. F., Dept. of Geology, The University, Bristol 8, England.
*Wickman, Frans E., Stockholm 50, Sweden.
Wiik, Birger, Fredsgatan 11-C-42, Helsinki, Finland.
Wilcox, Ray E., Geologic Division, Denver Federal Center, Denver 14, Colorado.
Wild, George O., Idar Oberstein 2, Germany.
Wild, Herman, 20243 Joann, Detroit 5, Michigan.
Wilkerson, Albert S., Dept. of Geology, Rutgers University, New Brunswick, New Jersey.
Willems, H. W., 1513 Pacific Avenue, Brackenridge, Pennsylvania.
Williams, Norman C., 1801 East 3900 South, Salt Lake City 7, Utah,.
Willis, Willett R., 1343 North Nevada Avenue, Colorado Springs, Colorado.
Willow, Stephen B., 2 West 47th Street, New York, New York.
Wilson, A. J. C., University College, Cathays Park, Cardiff, South Wales, Great Britain.
Wilson, Carl, 90 Tower Hill Road, Mountain Lakes, New Jersey.
Wilson, Ford E., 11844 S.E. Pine Street, Portland 16, Oregon.
*Winchell, Alexander N., 88 Vineyard Road, New Haven, Connecticut.
*Winchell, Horace, Geology Department, Yale University, New Haven, Connecticut.
*Winkler, H. G. F., Mineralogists Institut, (20b) Göttingen, Lotzestr. 16-18, Germany, British Zone.
Wintringham, Neil A., 50-10B Brookside Avenue, Somerville, New Jersey.
Wishart, James S., 56 Hammond Street, Rochester 13, New York.
Withrow, James R., 77 16th Avenue, Columbus, Ohio.
Wittels, Mark C., 120 West Hunter Circle K, Oak Ridge, Tennessee.
*Wolfe, C. Wroe, Dept. of Geology, Boston University, Boston, Massachusetts.
*Wood, Mrs. Elizabeth Armstrong, Bell Telephone Laboratories, Murray Hill, New Jersey.
Wood, Leonard E., 905 South Limestone, Lexington, Kentucky.
*Woodford, Alfred O., 443 West 10th Street, Claremont, California.
Woodhouse, C. D., 4125 Creciente Drive, Santa Barbara, California.

WOODRUFF, T. L., 3480 Morganford Road, St. Louis 16, Missouri.
WOODS, ARTHUR E., 1342 W. Beach, Pass Christian, Mississippi.
WOODWARD, THEODORE, 4207 Hewitt Avenue, Silver Spring, Maryland.
WOYSKI, MRS. M. W. SKILLMAN, Chemistry Department, University of Wisconsin, Madison, Wisconsin.
*WRIGHT, FREDERICK E., 2134 Wyoming Avenue, N.W., Washington, D. C.
WRINCH, DOROTHY, Dept. of Physics, Smith College, Northampton, Massachusetts.
WYATT, MICHAEL, St. Johns College, Cambridge, England.

YAGI, KENZO, Geophysical Laboratory, 2801 Upton St., N.W., Washington 8, D. C.
*YAGODA, HERMAN, Industrial Hygiene Research Laboratory, National Institute of Health, Bethesda 14, Maryland.
YEDLIN, LEO N., 557 W. Penn Street, Long Beach, New York.
YODER, HATTON S., JR., Geophysical Laboratory, 2801 Upton Street, N.W., Washington 8, D. C.
YORKS, KENNETH P., 104 Ormsby Drive, Syracuse 4, New York.
YOUNG, JOHN A., JR., Sun Oil Company, Production Dept., 1608 Walnut Street, Philadelphia 3, Pennsylvania.
YOUNG, ROBERT SPENCER, 426 East Buffalo Street, Ithaca, New York.
YOUNG, W. FORD, P.O. Box 102, Belize, British Honduras.
YU, PEI LIANG, 234 South Allen Street, State College, Pennsylvania.

ZAK, LUBOR, Mineralogical Department, Charles University, Prague 2, Czechoslovakia.
ZANNARAS, J. P., Box 500, Congress, Arizona.
ZEIHEN, LESTER G., c/o Chile Exploration Company, Chuquicamata (via Antofagasta), Chile, S. A.
ZEMAN, FREDERICK D., 111 East 88 Street, New York, New York.
ZEN, E-AN, 42 Kirkland Street, Cambridge 38, Massachusetts.
*ZERFOSS, SAMUEL, Crystal Section, Naval Research Laboratory, Washington 20, D. C.
ZIEDNER, MARTIN A., Box 1628, New Orleans, Louisiana.
ZIMMER, PAUL W., General Delivery, Crosby, Minnesota.
ZODAC, PETER, Box 29, Peekskill, New York.

MINERALOGICAL SOCIETY (LONDON)

The anniversary meeting of the Society was held on Thursday, November 1st, 1951, in the apartments of the Geological Society of London, Burlington House, Piccadilly, W.1 (by kind permission).

Exhibits

(1) Dalyite ($K_2ZrSi_6O_{15}$), a new mineral from Ascension Island (Atlantic); by Prof. C. E. Tilley on behalf of Dr. R. van Tassel.
(2) Silica glass from a crater in the western Sahara; by Dr. W. Campbell Smith.
(3) Adamite from Ojuella mine, Mapimi, Durango, Mexico; by the Department of Mineralogy, British Museum (Natural History), on behalf of Mr. D. E. Mayers.

Papers

The following papers were read:

(1) On the occurrence of varlamoffite in Cornwall.

By Sir Arthur Russell, Bart., and Dr. E. A. Vincent.

(2) A note on the occurrence of columbite in the Meldon aplite, Devonshire.

By Mr. Oleg von Knorring.

Small (0.3 mm.) tabular crystals, associated with tourmaline, apatite, topaz, fluorite, etc., were identified by *x*-rays and the predominance of niobium determined spectrographically.

(3) *X*-ray data from artificial bredigite, αC_2S.

By Mrs. A. M. B. Douglas.

Single crystal *x*-ray work has been done on material extracted from spiegeleisen slag. As suggested by Bredig, the structure is similar to, but is not identical with, orthorhombic βK_2SO_4 and a detailed analysis is in progress. Twinning on (110) has been confirmed. In some crystals a small amount of a trigonal phase, probably α C_2S has been found to co-exist with bredigite, and in one crystal a monoclinic phase was also found. The relative orientations of the trigonal, orthorhombic and monoclinic lattices in this crystal are in agreement with a simple mechanism of phase transformation.

(4) The melilite group of minerals.

By Dr. H. G. Midgley.

Calculation of the atomic ratios of the constituent atoms in some artificial melilites shows that they may be regarded as members of an isomorphous series with the general formula $(Ca, Na, K)_2(MgFe^{\cdot\cdot}Fe^{\cdot\cdot\cdot}Al)(SiAl)_2O_7$.

(5) The ledmorite dike of Achmelvich, near Lochinver, Sutherland.

By Dr. P. A. Sabine.

The presence of a dike cutting Lewisian gneiss near Achmelvich was recorded in the Geological Survey Memoir upon the Geological Structure of the north-west Highlands of Scotland and shown upon the Geological Survey maps. Re-examination of the dike has shown it to have affinities with the nepheline syenites. Its field occurrence and petrography are described and an analysis is given of the rock, which is classed as a ledmorite. There is little doubt that the dike belongs to the post-Cambrian pre-thrust-movement period of igneous activity of Assynt.

The following papers were taken as read:

(1) On crossite from Anglesey.

By Dr. N. Holgate.

A more detailed study of the Anglesey "glaucophane" than has previously been attempted shows that in chemical constitution and in optical properties it has more in common with the crossites than with any other of the amphiboles of the glaucophane-riebeckite series. Although the chemical analysis reveals some four per cent of lime, it is believed that the mineral is best described as a crossite, especially as all analysed crossites show some degree of "contamination" of this kind.

(2) The "superposition error" in the micrometric analysis of rocks.

By Dr. R. B. Elliott.

Where a mineral of high relief overlaps one of low relief in thin section, the low relief mineral is unnoticed and only the high relief mineral is seen. In micrometric analysis, therefore, high relief minerals will be overestimated and an error introduced. The amount of over-estimation is called the "error of superposition." Calculations are made, and graphs drawn, to relate the error to grain-size, making the assumptions, for mathematical simplicity, that the grains are orientated cubes. The error increases rapidly with diminution in grain-size and when the cubes are 0.03 mm. it is 100%.

(*Titles and abstracts kindly submitted by G. F. Claringbull, General Secretary.*)

German Meetings and Excursions in 1952

The 30th annual meeting of the German Mineralogical Society will take place in Regensburg between August 1–5, in connection with which a 3- to 4-day excursion has been planned through the Oberpfalz Forest and adjacent region including the localities: Wackersdorf (brown coal with a layer of pure, high temperature cristobalite), Wölsendorf (fluorite-barite veins), Amberg-Sulzbach (brown iron ore, weinschenkite, etc.), Hirschau-Schnaittenbach (kaolin), Hagendorf-Pleystein (feldspar pegmatite with triphylite alteration and rose quartz), and the Bayerland pit (pyrrhotite).

The German Geological Society will gather for its annual meeting August 10–15 in Osnabrück. An excursion is scheduled for August 9

The Swiss Mineralogical-Petrographical Society meets August 23–25. Very likely an excursion of several days will precede the gathering.

Geologists and mineralogists who are planning to participate in the International Geological Congress in Algeria from September 8–15, with the advance excursion beginning in Marseille on August 24 and whose itineraries will take them through Germany and Switzerland may wish to take part in the above listed meetings. They should contact respectively: (1) Prof. Strunz, Regensburg, Germany; (2) Prof. Stille, Hannover, Germany; (3) Prof. Galopin, Geneva, Switzerland.

NOTES AND NEWS

NOTES ON THE STAINING OF POTASH FELDSPAR WITH SODIUM COBALTINITRITE IN THIN SECTION

F. Chayes, *Geophysical Laboratory, Washington, D. C.*

Abstract

Several simplifying modifications of the staining procedure are described. The behavior of soda-rich or minutely perthitic potash feldspars is anomalous but the stain is highly selective for microcline in the presence of plagioclase and will not obscure perthitic intergrowths coarse enough to be readily resolved in unstained thin sections. The stain greatly facilitates both modal analysis and ordinary petrographic examination.

The staining of potash feldspar by sodium cobaltinitrite was suggested by Gabriel and Cox (1929) and extensively used by Keith (1939*A* and *B*), who combined it with an aniline dye stain for felspathoids in his study of the "leucolitchfieldite" of Blue Mountain, Ontario. The stain takes excellently on the potash feldspar of most granites. It eliminates any possibility of confusing potash feldspar with untwinned acid plagioclase and materially reduces the time spent in modal analysis. Even where questions of identification and analysis are not involved, a satisfactory stain greatly facilitates study of the habit and distribution of potash feldspar in the slide. In the course of the last three years I have stained well over 300 thin sections of calcalkaline granites and have made a number of minor revisions in the technique of Keith and of Gabriel and Cox. These revisions are hardly of fundamental importance but they considerably simplify and shorten the staining operation. They are described here in the hope that a more general appreciation of the simplicity of the technique and the elegance of the result will encourage petrographers to make staining a standard procedure in the study of granitic rocks.

Uncovering Old Slides for Staining

It is easier to work with new slides which have never been covered, but it sometimes may be desirable to uncover old ones. If the balsam is firm and hard the cover slip is easily sprung off with a razor blade. If the balsam is tacky the slide should be baked at about 80° C. until the tackiness disappears. In warm weather it is sometimes helpful to immerse the slide in cold water during removal of the cover slip.

After the slip has been removed, excess balsam is dissolved off the rock surface with xylol and alcohol. Gentle brushing with xylol for a minute, followed by brushing and rinsing in alcohol for two or three minutes, is usually sufficient. The surface will be sticky when the slide is removed from the xylol but should be firm and clean when it is taken from the alcohol. Following the alcohol rinse the slide should be washed thoroughly in running water. Any remnants of the covering medium still persisting may be removed by gentle rubbing with a little rouge or fine alundum, and a poorly stained slide may be prepared for retreatment in the same fashion.

At nearly every step of the uncovering procedure the slide is in grave danger; the first few slides are almost certain to suffer serious damage and may be lost. After a little practice, however, losses during uncovering are negligible.

THE HF ETCH

A lead bath is usually prescribed for the HF etch, but a plastic container is cheaper, lighter, and easier to keep clean. The old-fashioned square-shouldered butter dish sold in most 5-and-10-cent stores for very little more than 10 cents works admirably. The top serves as the bath. The base, on which the butter ordinarily is set, is used as a lid. A rack or bridge which will support the specimen tray is easily fashioned out of other plastic novelties. At the time my apparatus was assembled the local dime store was featuring something called a "plastibasket," the center of which, severed from the rest with a wire clippers, made an excellent bridge. The specimen tray consists of a piece of 1/4 inch plywood cut to fit loosely into the bath. As near each end as possible a wood screw is mounted; the tray is lifted into and out of the bath by tweezers which hold firmly under the head of each screw. A better specimen tray could surely be designed but this one has served satisfactorily for more than two years and is still as good as new. The level of acid in the bath should always be well below the surface of the bridge.

The backs of the slides are covered, either by taping with a strip of cellophane or by painting with a thin solution of rubber cement in xylol, and the slides are brought up to the temperature of the bath, between 30° and 40° C. A one-minute etch at 30° is the equivalent of several minutes of exposure to fumes at room temperature, but if the temperature is much above 45° the balsam is quickly attacked and the slide is ruined. High temperatures will also melt the plastic bath. The slides are brought to the temperature of the bath or a little higher before treatment in order to hold condensation to a minimum; they should be dry when they enter the bath and not more than barely moist when they are withdrawn. Any droplets which have condensed on the surface should be allowed to dry before the slide is stained.

Three or four slides are placed *face up* on the specimen tray, the cover of the bath is removed, the tray is placed in the bath by tweezers applied to the wood screws, and the bath is immediately covered again. For most calcalkaline granites a 45-second etch is sufficient and in general the shorter the etch the more even the stain. Working with a hot etching bath and a long staining period, a 15-second etch is often adequate. An etch of more than 2 minutes is likely to bubble and discolor the balsam and usually leads to patchy, ragged staining. The micas, particularly biotite, may also be attacked, but they rarely stain properly. When the mounting medium is not attacked, a stain which takes the form of minute equant crystals rather than a smooth, even coating is a good indication of overetching. Proper etching usually produces a faint frosting of the exposed upper surface of the slide; this frosting is often iridescent and can be removed by gentle rubbing. There is no need to remove it, however, for it will be washed off with the excess staining solution.

If the slides have been backed with cellophane tape the backing is removed as soon as the etch is completed. If rubber cement has been used it is more easily rubbed off during the rinse which follows the staining.

The Cobaltinitrite Stain

Instructions for making up the sodium-cobaltinitrite solution are given in both papers mentioned above, but the reagent can be purchased in powder form and is readily soluble in water. Both Keith and Gabriel and Cox recommend saturated acetic acid solutions, but the acetic acid is unnecessary and concentrations far below the saturation point sometimes do a better job than saturated ones. Three or four grams of reagent should be taken up in 6 cc. of water. Heating is not necessary but hastens solution. The liquid is at first yellowish red but becomes intensely brown or almost black when all the salt is dissolved. It should always be used at or below room temperature. Hot staining solution reacts as soon as it hits the slide, before it can be properly spread over the surface. The result is an irregular, blotchy stain.

The staining solution is quite stable but can be made up readily as needed, does not seem to improve with aging, and deteriorates rapidly with use. Ten cc. will usually be enough for 25 or 30 slides and each portion should be discarded after it has once touched an etched surface. It is best applied with a medicine dropper. Five or six drops are discharged onto the etched surface and then spread evenly over it with a glass rod or the side of the dropper. The surface of the slide must not be scratched during this operation, for each scratch will later appear as an unstained streak. When the liquid has been spread evenly over the entire surface a fresh drop or two may be added from the dropper. The slides are now allowed to stand for at least five minutes but much longer exposures do no harm and may be essential if a short etch or a weak staining solution has been used. It is to be remembered that the surface contains only a minute amount of available potassium and that every precaution has been taken to *avoid* prompt reaction. Overlong staining may permit the solution to evaporate to dryness or to work under the rock surface; in either case washing is likely to be difficult and unsatisfactory. If large batches are treated, the first slide is usually ready for rinsing a few minutes after the last one has been treated.

The Rinse and the Finished Product

The potash stain is remarkably sturdy and the slides are best rinsed in a gentle stream of cold water from the tap. Excess reagent is quickly removed and the slide is ready to cover as soon as it is dry. If the slides have been backed with rubber cement instead of cellophane tape, this should be rubbed off during the rinse.

In a properly stained slide the bright lemon yellow color of the potassium compound is prominent in plane light, almost imperceptible under crossed nicols, and sharply confined to potash feldspar. It has no noticeable effect on the index, extinction or birefringence of the potash spar, nor does it obscure or conceal perthitic intergrowths large enough to be resolved readily in unstained sections. In most rocks it is an immense aid in identification, modal analysis, and ordinary petrographic examination.

Cryptoperthites and non-perthitic or sub-microscopically perthitic alkali feldspars do cause trouble. Sometimes, as in the Deloro, Ontario, granite, they refuse to stain at all or stain very unevenly despite rather severe treatment. Sometimes, as with the pulaskite of Granite Mountain, Arkansas, they show little or no response to the ordinary procedure, but with more severe treatment take a smooth even stain which completely obscures the cryptoperthitic texture. My experience with rocks of this sort is still very limited but I am confident that much useful information would be generated by a careful study of the staining reactions of alkali feldspars, whether in plutonic or volcanic rocks.[1]

Such a study would be impossible without an abundant supply of first-grade sections, and in general it is one of the handicaps of work of this type that much wastage of thin sections is involved. Slight modifications of technique which lead to striking differences in sections may have no perceptible effect on sawed or polished tablets examined under reflected light.

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[1] Two modifications of the procedure, developed after this note was submitted to the journal, have eliminated most of the difficulty with cryptoperthite and alkali feldspar. The technique described above is adequate whenever the albite content, dissolved or exsolved, is low. When much albite is present or suspected the slide should be completely immersed and *gently agitated* in the staining solution. Suitable agitation of the slide largely overcomes the fatiguing effect noted above, and the staining solution need not be discarded after each application. The immersion technique is so satisfactory that I have begun to use it in routine work on calcalkaline rocks. Agitation materially shortens the necessary staining period. A 30-second etch followed by a 2- or 3-minute stain will usually be ample, but the alkali feldspar of some volcanic rocks may require as much as 10 or 15 minutes in the staining bath.

The smoothness and resolution of the stain are also considerably improved if the surface of the slide is smoothed before etching; the slide should be handlapped on a glass plate with #600 alumdum for 2 or 3 minutes.

References

Gabriel, A., and Cox, E. P. (1929), A staining method for quantitative determination of certain rock minerals: *Am. Mineral.*, **14**, 290–292.

Keith, M. L. (1939), Selective staining to facilitate Rosiwal analysis: *Am. Mineral.*, **24**, 561–565.

——— (1939), Petrology of the alkaline intrusive at Blue Mountain, Ontario: *Bull. Geol. Soc. Am.*, **50**, 1795–1826.

SIMPLE FIELD METHOD FOR THE DETERMINATION OF PHOSPHATE IN PHOSPHATE ROCKS

LEONARD SHAPIRO, *U. S. Geological Survey, Washington, D.C.*

A convenient field test has been developed in the Geological Survey for the detection and semi-quantitative estimation of phosphate in phosphate rocks. The basis of the test is the yellow phosphovanadomolybdate color which has been used for the determination of phosphorus in a variety of materials (Mission 1908). Materials containing wavellite or pseudowavellite must first be decomposed in boiling sulfuric and nitric acids to make the test, but tests for apatite may be made by merely shaking the finely ground sample with the nitric acid solution of the reagent. Although the test was devised to distinguish between samples containing 5, 15 and 30 per cent P_2O_5, it was found capable of giving results to the nearest 5 per cent. Interfering ions such as arsenic are not normally present in sufficient quantity to need consideration. Occasionally the samples must be ignited to remove organic matter.

Apparatus

1 small porcelain mortar and pestle.
1 alcohol lamp.
1 measuring spoon—a small metal rod with a 1/16 inch hole drilled to a depth of 1/16 inch. Do not use a rod of plastic or other material which might become charged with static electricity.
3 reference solutions described below.
1 small dropper with rubber bulb.
3 test tubes marked at 15 ml., of the same size as that containing the reference solutions.
1 test tube clamp.

Reagents

Mixed acid; 15 ml. of concentrated nitric acid plus 30 ml. of concentrated sulfuric acid.

Vanadomolybdate reagent: Dissolve .300 g. of ammonium metavanadate in 200 ml. of water, and add 50 ml. of concentrated nitric acid. Dissolve 12.5 g. ammonium molybdate in 100 ml. of water. Add this to the vanadate solution and make to 500 ml.

Preparation of Reference Solutions

A known phosphate rock sample containing phosphate as apatite and a rock sample known to contain a negligible amount of phosphate are mixed in the proper proportion to give 5, 15 and 30 percent P_2O_5. For example, using Bureau of Standards Sample #120 (Tennessee phosphate 35.3% P_2O_5 and Sample #1A (limestone) take the following:

.3 g. #120 plus 1.7 g. #1A ≈ 5 per cent P_2O_5
.9 g. #120 plus 1.2 g. #1A ≈ 15 per cent P_2O_5
2.0 g. #120 plus .3 g. #1A ≈ 30 per cent P_2O_5

Grind each mixture together with the mortar and pestle, and carefully mix again. Then transfer a sample of each of these to test tubes, measuring the samples by volume by dipping the measuring spoon into the powder and sweep away the excess with a finger. Pour vanadomolybdate reagent into each tube to the 15 ml. mark. Stopper the tube with a cork and invert two or three times. After five minutes, the color is at a maximum and remains stable for several days. Permanent standards may then be made by diluting an aqueous solution of potassium dichromate in test tubes until there is a match between each dichromate solution and previously prepared standards. Fifteen ml. of each of the dichromate solutions is then sealed permanently in the test tubes, these will last indefinitely.

Test for Apatite

Crush a small quantity of the rock to an impalpable powder. Then shake a measured volume of the powder in a test tube with 15 ml. of vanadomolybdate reagent and compare with the permanent standards representing 5, 15 and 30 per cent P_2O_5.

Testing Samples Containing Wavellite and Pseudowavellite

Wavellite and pseudowavellite do not dissolve readily in cold dilute nitric acid and must first be boiled with concentrated sulfuric and nitric acids before testing. Apatite may thus easily be distinguished from wavellite and pseudowavellite.

Dip the measuring spoon into the sample, ground to an impalpable powder, to fill the hole in the measuring spoon; sweep away the excess with a finger, and transfer the contents of the spoon to a test tube. Add 10 drops of the mixed acid and heat over the alcohol burner for about a minute. Stop heating before the contents of the test tube go to dryness. Cool and add the vanadomolybdate reagent to the 15 ml. mark. Stopper the tube with a cork, shake, and compare with standards after 5 minutes.

Reference

Mission, G., Colorimetric estimation of phosphorus in steels: *Chem. Ztg.*, **32**, 633 (1908).

NEW LOCALITY OF SHORTITE

Max P. Erickson, *University of Utah, Salt Lake City, Utah*

Introduction

The mineral shortite has been identified in the drill core of the Sun Oil Company's Ouray Well located in Uintah County, Utah. It is believed to be the second known recorded occurrence of this mineral.

Shortite was originally described by Fahey (1) in 1939 from material found in a drill core from a well near Green River, Wyoming, about ninety miles due north of the Utah locality. It occurred in the Green River formation of Eocene age. The crystals are orthorhombic hemimorphic exhibiting forms $\{00\bar{1}\}$, $\{100\}$, and $\{011\}$, and have an axial ratio $a:b:c=0.455:1:0.648$. They are clear and colorless to pale yellow in color with conchoidal fracture and vitreous luster. Hardness is 3 and specific gravity 2.629.

Optical properties are recorded as $\alpha=1.531$, $\beta=1.555$, $\gamma=1.570$; $2V=75°$ (optically negative). Chemical analyses yielded the formula $Na_2CO_3 \cdot 2CaCO_3$.

The x-ray data were published by Richmond (2) in 1941.

Utah Occurrence

The Ouray Well is located in Sec. 22 T. 9 S., R. 20 E., Salt Lake Base and Meridian, approximately seven miles southeast of Ouray, Uintah County, Utah. The shortite is found in the Green River formation between the depths of 1975 feet and 2056 feet.

The mineral is found as individual crystals concentrated in bands varying from a fraction of an inch to several inches in thickness. It is associated occasionally with minor amounts of pyrite. The separate crystals are euhedral and exhibit forms apparently identical to those described by Fahey (3). The crystals are up to 15 millimeters in length and are colorless with a vitreous luster; their hardness is about 3. Maximum and minimum indices of refraction were determined by the immersion method as $\alpha=1.530$ and $\gamma=1.570$. The mineral is biaxial $(-)$ with a moderately large axial angle.

The mineral effervesces vigorously with dilute hydrochloric acid and the resulting solutions contain abundant sodium and calcium as determined by microchemical tests.

On the outside of the core, sections of the crystals cut in various crystallographic directions are quite conspicuous. The exposed surfaces of these crystals consist of a fine aggregate of pink colored calcite. This is apparently a result of the solution and removal of sodium carbonate by the water in the drilling mud and recrystallization of the residual calcium carbonate to form a fine aggregate of calcite. This effect penetrates less than one millimeter into the crystals. The crystals in the interior of the core are clear and unaffected.

References

1. Fahey, Joseph J., Shortite, a new carbonate of sodium and calcium: *Am. Mineral.*, **24,** 514–518 (1939).

2. Richmond, W. E., *X*-ray crystallography of shortite: *Am. Mineral.*, **26,** 288–289, 629–630 (1941).
3. Fahey, Joseph J., *op. cit.*, 515–516.

Condensed Interim Report of the American Geological Institute

The annual meeting of the American Geological Institute was held in Detroit, Michigan, on November 6 and 7. Officers elected for the coming year are:

President..........Carey Croneis
Vice-President..........Joseph L. Gillson
Secretary-Treasurer..........Harry S. Ladd

A number of useful reports and publications are currently planned for the near future. Some of these will be new; others will be re-issues of previous reports which are now outdated. They are:

(*a*) A directory, "Departments of Geological Science in Educational Institutions of the United States and Canada." Previously issued by A.A.P.G. in 1949. It is proposed to issue this Directory on an annual basis hereafter.

(*b*) "Guide to Societies of Geological Science in the United States and Canada." This directory is planned for early in 1952. It will contain the names of officers and councilors, major publications and other pertinent information concerning each society.

(*c*) Geological Guide to U. S. Highway 75 from Galveston, Texas, to the Red River. This report, being prepared by F. W. Rolshausen of Houston, Texas, will provide not only geological but also tourist information, geological maps, type sections and other information of use to the traveling geologist. To be issued in the spring of 1952. This is intended to be a "type specimen" for a number of others planned, or under discussion.

(*d*) A "career booklet" on geological science is being planned for issuance in the spring of 1952. No booklet of this type is currently available, so that vocational advisors lack information concerning the profession. Statistics from the National Scientific Register concerning the structure of the profession will be included.

(*e*) The directory of "Summer Geology Field Courses," first issued in the spring of 1950, will be re-issued in February or early March of 1952. Information for this report is being requested from departments during December.

(*f*) Non-Industrial Research in the Geological Sciences, first compiled in early 1950, will be brought up-to-date and re-issued in May or June of 1952.

(*g*) A new and up-to-date edition of the Catalogue of Geological Guide Books and Road Logs, issued in 1950, is now in preparation and should be available in February, 1952.

The Committee on a Glossary of Geological Terminology is proceeding vigorously to compile its material and is making good progress under the leadership of J. V. Howell of Tulsa, Okla. Investigation has revealed the existence of not less than fifteen unpublished specialized glossaries which are available to the Committee. This will greatly reduce the work of the Committee and speed the final compilation of the volume.

John Putnam Marble
Director from the Mineralogical Society of America

John J. Forbes, a long-time employee of the Bureau, was named Director of the U. S. Bureau of Mines, succeeding James Boyd who resigned to become vice-president of the Kennecott Copper Co. Mr. Forbes holds a degree in mining engineering from Pennsylvania State College and has been with the Bureau since 1915.

Paul Zinner, formerly regional director of Region V at Minneapolis, has been appointed to the post of Chief of the Minerals Division in Washington, D. C., and Paul T. Allsman, who has been with the Denver office of the Bureau, will fill the regional directorship at Minneapolis, vacated by Mr. Zinner.

Three geologists received awards for outstanding work during the 64th annual meeting of the Geological Society of America and associated societies in November 1951. Recipient of the Penrose medal of the Geological Society was Pentti Eskola, petrologist from the Geological Institute of Finland; Martin J. Buerger, professor of mineralogy and petrography at the Mass. Institute of Technology, received the Geological Society's Arthur L. Day medal; and Orville F. Tuttle of the Geophysical Laboratory, Carnegie Institution of Washington, was the first recipient of a new award of the Mineralogical Society of America.

A bibliography of loess containing some 280 citations has been prepared by the staff of the Iowa Engineering Experiment Station in connection with a research project on the loess and glacial till materials of Iowa. Copies of the bibliography may be secured without charge by writing to the Director, Iowa Engineering Experiment Station, Ames, Iowa.

Tentative Program for a National Conference on Clays and Clay Technology, University of California, Berkeley, California

July 21–25, 1952

Monday, July 21st, 9–12 A.M. Session

Introduction

1. Introduction—Definitions—Objectives
2. Mineralogy
3. Origin, Occurrence and Fabric of Clays

Monday, July 21st, 2–5 P.M. Session

Panel Discussion

Tuesday, July 22nd, 9–12 A.M. Session

Properties

4. Electrokinetic and Electrochemical
5. Ion Exchange
6. Adsorptive and Swelling Properties—Water Systems
7. Adsorptive and Swelling Properties—Organic Systems

Tuesday, July 22nd, 2–5 P.M. Session

Panel Discussion

Evening

Conference Dinner

Wednesday, July 23rd, 9–12 A.M. Session

Identification Methods and Their Interpretation

8. Particle-Size Distribution
9. Chemical Analysis
10. Petrography
11. Dye Adsorption
12. Infrared Absorption

Wednesday, July 23rd, 2–5 P.M. Session

Identification (Continued)

13. *X*-Ray Diffraction Analysis
14. Electron Microscopy
15. Differential Thermal Analysis

Panel Discussion

Evening

Panel Discussion (Continued)

Thursday, July 24th, 9–12 A.M. Session

Clay Technology in

16. Soil Science
    a. Physical aspects
    b. Chemical aspects
17. Soil Mechanics
    a. Physical aspects
    b. Chemical aspects
18. Ceramics

Thursday, July 24th, 2–5 P.M. Session

Panel Discussion

Evening

Visits to Laboratories

Friday, July 25th, 9–12 A.M. Session

Clay Technology in (Continued)

19. Fillers
20. Bleaching and Filter Clays
21. Petroleum Industry
    a. Drilling fluids
    b. Reservoir problems
    c. Refining

Friday, July 25th, 2–5 P.M. Session

Panel Discussion

Further information can be obtained from Professor J. E. Pask, Division of Mineral Technology, University of California, Berkeley 4, California.

PEACOCK MEMORIAL PRIZE,* 1952

Offered by the

WALKER MINERALOGICAL CLUB

Royal Ontario Museum of Geology and Mineralogy,
100 Queen's Park, Toronto 5

THIS PRIZE of ONE HUNDRED DOLLARS cash is again offered for the best scientific paper on pure or applied mineralogy (including crystallography and petrology) submitted to the Club by a graduate student at any university or similar institution.

Conditions

1. The papers submitted may offer new or refined observations, or a significant synthesis and interpretation of existing data, or some new or improved application of mineralogy to useful ends, or the results of other work of sufficient interest and value.

2. The papers should be typed, double-spaced on letter-size paper and illustrations should be carefully prepared and suitable for reproduction. References to the literature should follow the style in *Contributions to Canadian Mineralogy* as printed in *The American Mineralogist* (May–June issues, 1949–50–51).

3. Each paper must be accompanied by a letter from the student's supervisor stating the nature and extent of the assistance he may have given to the work submitted.

4. The papers are to be sent to the Secretary, Walker Mineralogical Club, 100 Queen's Park, Toronto, *not later than June 30, 1952.* They will be judged by a committee appointed by the Council of the Club who will consider both the value of the papers and their suitability for publication without substantial changes. If no paper of sufficient merit is received, the prize will not be given.

5. The Committee will select one or more of the papers for presentation by the authors, or other designated persons, at a regular meeting of the Club, and the result of the contest will be announced at that time.

6. The prize winning paper, and perhaps others of sufficient merit, will be published in *Contributions to Canadian Mineralogy.*

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* Instituted in 1947 as the Walker Mineralogical Club prize, was renamed the Peacock Memorial Prize in 1950 in honour of the late Professor Martin A. Peacock, President of the Walker Mineralogical Club 1942–43, and Editor of Contributions to Canadian Mineralogy, 1944–50.

Prizes have been awarded to:

A. R. Graham, Queen's University, (1947), "*X*-ray Study of Chalcosiderite and Turquoise": *Univ. Toronto Studies, Geol. Series,* No. **52,** 1948. (Contributions to Canadian Mineralogy, Volume 4, No. 4.)

I. H. Milne, University of Toronto, (1948), "Chloritoid from Megantic County, Quebec": *Am. Mineralogist*, **34,** 1949. (Contributions to Canadian Mineralogy, Volume 5, No. 1).

R. J. Arnott, University of Manitoba, (1950), "*X*-ray Diffraction Data on Some Radioactive Oxide Minerals": *Am. Mineralogist*, **35,** 1950. (Contributions to Canadian Mineralogy, Volume 5, No. 2).

ANNUAL MEETING

The thirty-third annual meeting of the Mineralogical Society of America will be held in Boston, Massachusetts, on November 13–15, 1952, with headquarters at the Hotel Statler.

A series of field trips is being planned for Monday, Tuesday, and Wednesday, November 10th to 12th. Various short excursions to scientific laboratories, industries, and historical sites in the Boston area are planned for the days of the meeting, November 13th–15th.

Abstracts of papers to be presented at the annual meeting must be received by the Secretary on or before *July 15, 1952*. Abstract blanks may be obtained from the Secretary.

C. S. HURLBUT, JR., *Secretary*

The Geology and Mineralogy Section of the Michigan Academy of Science, Arts and Letters held its annual meeting at the University of Michigan, Ann Arbor, Michigan on Friday, April 11. Papers were presented at two sessions: A morning meeting devoted to varied subjects and an afternoon meeting consisting entirely of papers on the iron deposits of Michigan and allied problems in Michigan pre-Cambrian geology.

Professor J. C. Griffiths has kindly called attention to computation errors in Table 5 of our article, "A Test of the Precision of Thin-Section Analysis by Point-Counter" (this journal, 1951, pp. 704–712). In Table 5 (p. 710) the values for muscovite should be 0.1744 under the column headed "Analysts"; 0.1504 under the column headed "Slides"; and 0.1101 under the column headed "Error (obs.)." The excess of observed over expected error is still highly significant so that the corrections require no change in our interpretation of the table.

FELIX CHAYES AND HAROLD W. FAIRBAIRN

Corrections

Vol. **37** (1/2) Jan.–Feb. 1952:

Page 104, base; γ in (31′) should be α.

Page 105, lines 10 and 22; *diffraction* should read *precession*

Page 109, Table 6, last line, right column; prime the G_2.

BOOK REVIEWS

THE INTERPRETATION OF *X*-RAY DIFFRACTION PHOTOGRAPHS, by N. M. F. Henry, H. Lipson and W. A. Wooster, MacMillan and Co., London, 1951, ix+258 pages, price 42 shillings. (American price $8.50, obtained from D. Van Nostrand Co., Inc., New York, N. Y.)

This book is intended "to help students and research workers to understand the theory and practice of the interpretation of *x*-ray diffraction photographs." The discussion is carried up to the subject of crystal-structure determination but does not include more than passing reference to space groups and their recognition by means of *x*-ray diffraction. Had a chapter on space groups been included, and it would have required no more space than that devoted to Laue photographs in the present volume, all materials needed preparatory to crystal structure work would have been brought together. The omission is the more regrettable because the authors state that "a sufficient number of tables has been included to facilitate calculations without the necessity of a reference book." Many who do not embark upon crystal structure work do attempt the determination of space groups and a statement of the space group, now considered part of a complete mineral description, is to be found in several recently published mineralogical reference books.

What seems to this reviewer the pedagogical excellence of Henry, Lipson and Wooster's book is probably due in part to the fact that the authors have been associated with the very thorough course in crystallography which has been offered at the University of Cambridge for many years. The book has many fine features. It is clearly written and very well illustrated. There are diagrams on almost every page and many points are illuminated by detailed discussion of examples. The reciprocal lattice is used extensively. "Much material is presented in a geometrical way, as it has been found that this is the most suitable method of teaching crystallography." A few problems for students are included and carefully worded answers are given in an appendix. A pleasing and novel feature is the reference list which has been arranged to serve as a name index. An appropriate statement about the relation between kX and Å units is made in chapter 2. Nevertheless kX units are called Å units in the first ten chapters, while Å units proper are used in chapters 11 to 17. It is to be feared that this will perpetuate the existing confusion as few will read or remember the authors' note explaining the curious manner in which they have dealt with this problem.

The emphasis is obviously determined by British conditions and especially by the experience of the authors. This will restrict the usefulness of the book in the United States. Charts for the interpretation of single crystal patterns are based on a camera radius of 3 cm. By photographic reduction they may be adapted to the 57.3 mm. diameter commonly used. The scant space given to important modern techniques widely used in this country is a lack not so easily overcome. The precession method is barely mentioned and the equi-inclination Weissenberg method receives only 2 paragraphs of discussion while many pages are devoted to methods that will be considered obsolete by some.

Gypsum is used as an example in a number of chapters and finally in chapter 10 the problem of transformation of axes and face indices is illustrated by gypsum. Here the authors have neglected to consider the problem of the "right setting" and have mentioned only three of the many settings that have been used for gypsum. Among these the oldest setting, that of Haüy, and the setting corresponding to the shortest possible *a* and *c* axes are not included. The authors have here missed a fine opportunity to exploit fully the example they have used in several chapters.

The shortcomings of this book will not seriously impair its value to those many people who have need to acquaint themselves with the subject it treats. It is to be hoped that a new edition will soon be called for and that the authors will then bring the book fully up to date. In the meantime this first edition can be heartily recommended to anyone requiring a clear explanation of the use of x-ray diffraction photographs.

A. Pabst,
University of California, Berkeley 4, California

GEOLOGIE, MINERALOGIE UND LAGERSTÄTTENLEHRE, by Paul Kukuk, vii+306 pages; 370 figs., Springer Verlag, Berlin, 1951. Price, DM 28.50.

This book is designed to fill the need for brief yet comprehensive German textbooks of geology, mineralogy and ore deposits. It is intended primarily for pupils and teachers in mining and technical schools, but mining engineers, mine surveyors and officials, and others interested in mining geology will find it helpful.

Part I is a 137-page treatment of the fundamentals of physical and historical geology. The section on physical geology is introduced by a very brief discussion of the structure of the earth and isostasy. Twenty-four pages are devoted to a general description of igneous rocks, sedimentary rocks, and the "crystalline schists," with another 16 pages devoted to rock structure. The Rosenbusch classification of igneous rocks is followed, even to giving different names to volcanic rocks of the same composition according to whether they are pre-Tertiary or younger. A very simple descriptive classification of sedimentary rocks is used and only a few of the more important types of metamorphic rocks are mentioned.

Ground water and springs are briefly discussed at the end of the section. Geological forces and their operation are classified into surface and "inner" phenomena, and merit a 41-page discussion.

In the section on historical geology there is a 2-page synopsis of the plant and animal kingdoms and a 2-page tabular summary of geologic history giving eras, periods, approximate ages in years and the development of plant and animal life. There follows a 37-page cursory outline of the geologic periods with 40 illustrations of typical sedimentary rocks and fossils.

Part II is a 73-page treatise on mineralogy. Crystallography is allotted but 3 pages. All of the crystal systems are illustrated with axes and angles, but the more important crystal forms are given for the isometric system only. Physical and chemical properties of minerals, origin of minerals, and crystal structure are given very little space. Optical properties are not discussed, and origin of minerals is summarized on a single page. The major part of this section is given to some 90 descriptions of species of rock forming, gangue and ore minerals in more or less detail, with respect to composition, properties, occurrence and use. About 25 others are mentioned but not described.

Part III, the economic geology section, consists of 86 pages of description and discussion of the coal, ore, salt, and oil deposits of Germany, giving geographical distribution, external form, relation to country rock, genesis, use and estimates of reserves. Geophysical methods of prospecting are outlined in slightly less than two pages.

At the end of the section occurrences of industrial minerals (graphite, fluorite, barite, gypsum, magnesite, etc.), building stones, clay, natural gas, and precious stones are mentioned very briefly.

The work of many authors is referred to in the text, but references are limited to a single page at the end of the book of the more important sources of material. There is a 10-page subject index.

Kukuk certainly attained his objective of presenting briefly and concisely a tremendous amount of information. Most sections are probably adequately treated for the audience to which the work is addressed, but several subjects are touched so lightly that it appears to the reviewer they might better have been expanded or omitted entirely. For example, the section on crystallography would have been much more useful if the symmetry and principal forms of the other systems had been outlined; this would have required only 3 or 4 pages more to treat them the same way the isometric system is described. There is little point to discussing crystal structure and *x*-ray patterns in a work of this kind; one page is certainly too little space to give any useful idea of these subjects. It would seem that in a book in which so much emphasis is placed on useful mineral deposits more than two pages could have been profitably devoted to geophysical prospecting, and geochemical prospecting might have been mentioned.

Neither the preparation nor interpretation of topographic or geologic maps is mentioned in the part on geology. However, simple geologic maps of some of the German mining districts are given in the part on ore deposits.

Part I (geology) emphasizes processes and other information of interest to mining men, engineers, and other practical scientists, which probably explains the omission or extremely brief treatment of many subjects to which considerable attention is given in most textbooks of geology. For example, the only reference to the ocean is a one-page discussion of marine abrasion and the only reference to deserts is by implication in the outline of the work of wind. Soil creep and landslide phenomena are not mentioned at all.

The book is very well illustrated with excellent photographs and diagrams. It is well bound in cloth and should prove to be a compact and handy reference work for mining men as well as a textbook for students of mining geology and related branches of earth science. It touches a wider range of subjects than do most textbooks of geology and so should prove useful to non-Germans in acquiring a larger vocabulary of German terms by reading of a single small volume.

Earl Ingerson,
U. S. Geological Survey, Washington, D. C.

GUIDE TO GEOLOGIC LITERATURE, by Richard M. Pearl, xi+239 pages, McGraw-Hill Book Co. Inc., New York, N. Y., 1951. Price, $3.75.

The scope of Pearl's book is a good deal broader than is indicated by the title. After a brief introduction (Part I, 13 pp.) in which the problem is stated there is a 33 page treatment of library facilities (Part II) with chapters on Arrangement of the library, Library catalogues, Indexing and Library services. Most of this information is of a general nature and applies to any kind of library reference. However, library catalogues of special interest to geologists and libraries well-known for geologic and geographic research are listed specifically, although most of these libraries are among the most important for a wide variety of subjects.

Part III is a 146-page discussion of the kinds of geologic literature. There are 13 chapters, each of which is devoted to a type of publication. Most of these types are well recognized, but some of them, such as newspapers and city and county bulletins are not commonly thought of as sources of geologic material. In most of the chapters of Part III, especially those on Index guides and bibliographies, Abstracts, and Periodicals, much information is given about the literature of chemistry, physics, geography, agriculture, general science, and other subjects even further afield from geology.

Interesting and useful information about federal and state agencies, which one would hardly expect to find in a book on geologic literature is included in the chapters on the publications of those agencies. For example, an organization chart and list of regional headquarters is given for the U. S. Bureau of Mines (but not for the U. S. Geological Survey); the regional offices of the Soil Conservation Service (Agriculture) are given; the divisions of the National Bureau of Standards and its series of publications (most of which have very little to do with geology) are listed; all state and territorial geological surveys, bureaus of mines, and agricultural experiment stations are listed with their addresses and additional information is given about the first two.

This breadth of treatment makes the book especially useful to those geologists who are not well acquainted with the literature of the related natural sciences or with some of the more obscure types of geologic or "borderline" material. However, the inclusion of so much extraneous material, rambling treatment, and repetition combine to make the book much longer than appears really necessary for a treatise of this kind. It also suffers from lack of organization, poor definition, hazy classification, and inaccuracies. Much of the reference material would have been more useful had the presentation been in list form, with notes, rather than being scattered throughout the text.

Of the inaccuracies and omissions noted the following appear to be of sufficient interest to merit mention in a review.

On p. 84 the information about volume numbers of the *American Journal of Science* is incomplete; after vol. 36 of Series 5 there is a break, the next volume going to the "whole number" 237 (1939), and since then the volumes have been numbered consecutively one per year instead of 2 per year as previously. The whole volume number of the reference near the bottom of the page should be 236, not 216.

Similarly the information (pp. 125–126) on the volumes and numbering of the *Transactions of the American Geophysical Union* is in error. Since 1945 (Vol. 26) they have been issued as a regular bimonthly journal. Before that time they were not assigned volume numbers, but were designated as reports of annual meetings.

On p. 125 the statement that "N.R.C. Committee Reports are not available for general distribution," is erroneous because many of the longer and more useful reports such as the one issued each year by the Committee on Geologic Time are for sale to anyone interested. On p. 203 the address of the National Geographic Society should be Washington, D. C., instead of New York.

The coverage of this book is so broad and comprehensive, that the omission of several better known reference works is all the more striking. In the treatment of bibliographies it probably would have been well to list at least one on petroleum, for example, De Golyer and Vance, *Bibliography on the Petroleum Industry*, Texas A. and M. College, Bull. 83,730, p., 1944. Also Mark Pangborn's *The Earth for the Layman*, Amer. Geol. Inst., Rep. No. 2, 50 p., Washington, 1950, would be a useful addition. Although Lahee's *Field Geology* (McGraw-Hill, N. Y., 1941) is not called a handbook in its title it certainly is one in content and treatment and therefore should have been included in the list of such books on p. 171. It probably would have been worthwhile to list on p. 90 the 26 most important geologic periodicals referred to there. In the same chapter the new journal *Geochimica et Cosmochimica Acta* might also have been mentioned.

On p. 122 there is a list of publications of the National Bureau of Standards which omits their important *Journal of Research*. "Open file reports" form an increasingly important part of the out-put of the U. S. Geological Survey, yet these are not mentioned either under U. S. Government Documents or under "Unpublished Manuscripts." On pp. 174–175 there is a list of English-foreign-language geologic dictionaries; in the experience of the reviewer, other technical dictionaries, a few of which are given here, are more useful to and more widely used among geologists, although most of the titles do not indicate that geologic terms are included:

"German-English Dictionary for Chemists" by Austin M. Patterson, John Wiley and Sons, Inc., New York 3d ed., 1950, 541 pp.

"French-English Dictionary for Chemists" by Austin M. Patterson, John Wiley and Sons, Inc., New York, 1921, 384 pp.

"German-English Science Dictionary" by Louis De Vries, McGraw-Hill Book Co., New York, 1939, 473 pp.

"French-English Science Dictionary" by Louis De Vries, McGraw-Hill Book Co., New York, 1940, 546 pp.

"Engineer's Dictionary—Spanish-English and English-Spanish" by Louis A. Rabb, John Wiley and Sons, Inc., New York, 1949, 664 pp.

"Russian-English Technical and Chemical Dictionary" by Ludmilla Ignatiev Callaham, John Wiley and Sons, Inc., New York, 1947, 794 pp.

Recently three more good technical dictionaries have appeared that should be useful for geologists:

"German-English Technical and Engineering Dictionary" by Louis De Vries, McGraw-Hill Book Co., New York, 1950, 928 pp.

"Diccionario Minero-Metalúrgico-Geológico-Mineralógico-Petrográfico (English-Spanish-French-German-Russian) by Alejandro Novitsky, Buenos Aires, 1951, 309 pp. quarto.

"Tekniikan Sanasto (Technical Vocabulary, German-English-Finnish-Swedish-Russian)," Kustannusos akeyhtiö Otavan Kirjapaino, Helsinki, 1950, 1518 pp. (exclusive of index, which is a separate volume).

The analysis of unpublished research in geologic science mentioned on p. 193 has now been published as Report No. 3 of the American Geological Institute (Washington, D. C.) with the title "Non-Industrial Research in the Geological Sciences, 1950." It lists 3048 projects in 80 mimeographed pages. A companion volume, "Current Research in the Geological Sciences in Canada, 1950–51" compiled by J. F. Henderson, was published in Ottawa in 1951 by the Geological Survey of Canada. It has 54 pages and lists 548 projects.

Pearl's book has an unusually comprehensive index of 27 pages for 212 text pages. Some omissions, however, are inevitable. For example, there is no entry "Interlibrary loan" or "Loan, interlibrary" or "Library, loans" although the subject is treated in some detail. There are no index references to the British colonies listed at the bottom of p. 156.

Guidebooks to the literature of most of the other natural sciences have long been available. With the growing importance of Geology for the national economy and the increasing complexity of published material the appearance of *Guide to Geologic Literature* is very timely. Such a compendium will be valuable not only to students who are getting acquainted with the literature, but also to more experienced geologists who thought they already knew most of the field.

EARL INGERSON
U. S. Geological Survey, Washington, D. C.

GEOMETRISCHE KRISTALLOGRAPHIE UND KRISTALLOPTIK UND DEREN ARBEITSMETHODEN, by FRANZ RAAZ AND HERMANN TERTSCH (Second Edition), x+215 pages, Springer-Verlag, 1951. (Price, paper-bound, $4.50).

The first edition of this textbook (1939) appears to have been very well received by colleges in German-speaking countries, which calls for a revised edition. However, the second edition is practically a reprint of the first; table of contents, pagination, figures and index are practically identical in the two editions. Only very minor changes or additions have been made; for example, there are two three-line supplementary statements on p. x, and in the discussion of universal stage technique a reference to the excellent treatment of the subject by Berek has been added.

Part I is a 123-page straightforward treatment by Franz Raaz of geometrical crystallography (crystal morphology). Fundamental laws of crystallography, Methods of representing crystals graphically, Crystal symmetry, Development of the 32 crystal classes, Description of the forms in the individual crystal systems, Development of twins, and Crystal structure are discussed in that order.

Part II, by Hermann Tertsch, is an 85-page outline of crystal optics in which Fundamentals of the optical character of crystals, Polarizing microscopes, Crystals showing optical rotation, and Influence (of temperature, pressure, etc.) on the optical characters of cystals are discussed in that order.

This is a very compact, and for the most part accurate, textbook of crystallography and crystal optics. However, there are a number of things that might have been changed with advantage in a "revised" edition. Some of the illustrations (e.g. Fig. 24, an 1893 model of a two circle goniometer) are even further out of date than they were in 1939; there is little point to having two identical illustrations like Figs. 36 and 74. On p. 164, par. 5 the author fails to recognize that in a random section of a uniaxial mineral one direction always gives the true value of ω. In the first paragraph of p. 172 the U.M. series of objectives made by Leitz might have been mentioned. They have initial magnifications up to 30 X with the universal stage and in large part overcome the difficulty mentioned here. The directions for use of the universal stage on pp. 172–173 are simplified and abbreviated to the point of inaccuracy in some respects. For example, in par. 2, p. 172, the statement is made that if a mineral grain remains dark between crossed nicols during rotation about either horizontal axis it is isotropic. This is usually, but by no means always, the case. Grains, or sections, of uniaxial and biaxial crystals in certain orientations will behave in this manner. Likewise, the descriptions of measuring uniaxial and biaxial crystals are so generalized that they do not take care of all possible initial orientations. In the reviewer's opinion this section should either have been expanded considerably or just a reference given to adequate treatments of the subject.

The book is well illustrated, is printed on good paper and presents an over-all pleasing effect. Particularly attractive are the pictures showing crystal models, or drawings of crystals, alongside crystals of minerals showing the same form(s). The discussions of skiodromes and of Bertin surfaces are much more complete and satisfactory than those found in most English textbooks. The reviewer is completely in sympathy with Tertsch's use of ω and ϵ and α, β and γ to denote the indices of refraction of uniaxial and biaxial cyrstals, respectively. Used in this connection they are entirely unambiguous; if this usage could be made universal it would eliminate a great deal of uncertainty of meaning and awkwardness of expression in many American and English textbooks and articles.

EARL INGERSON,
U. S. Geological Survey, Washington, D. C.

ELEMENTS OF OPTICAL MINERALOGY. PART II—DESCRIPTION OF MINERALS, by A. N. WINCHELL WITH COLLABORATION OF HORACE WINCHELL, 4th Edition, 1951, viii plus 551 pp., 6 by 9 inches, 427 figs. Cloth. Price $12.50. John Wiley and Sons, Inc., 440 Fourth Ave., N. Y. 16, N. Y. The publishers have issued an errata sheet, free on request.

This book is a thoroughly revised edition of the well-known and widely used reference work of A. N. Winchell. According to the publishers, the volume of new data and descriptions have been expanded by about 25 per cent. There is an increase of 92 pages over the third edition. All those who use this book will be greatly indebted to the Winchells for a valuable, handy, and usable reference book that is indispensable in the laboratory.

The authors have combed the scientific literature so well that the book contains all of the latest data available to them at the time of preparation.

Having stated unequivocally the importance of this work to mineralogists, I now present a critique of its contents:

Immediately following the table of contents is a very useful summary of abbreviations and symbols. The statements on page xiv that:

N_x .... α of German writers
N_y .... β of German writers
N_z .... γ of German writers

apparently changes the nationality of many residents of the United States and Great Britain.

A *Special Notice* is printed on pages xv and xvi for users of this volume. First, there has been a complete change in symbolism for the indices of refraction—the N_g, N_m, N_p, N_o, and N_e are now replaced by N_z, N_y, N_x, N_0, and N_E. Although this is a very great concession on the part of the senior author, nevertheless, it establishes a new set of symbols. It is to be regretted that the authors did not accept one of the existing sets of symbols. Second, a very commendable usage has been introduced, in treating the data for extinction angles, by adopting Schuster's rule which heretofore has been followed largely in describing feldspar crystals.

The introduction to the book is an illustrated section on Drawings, Projections, and Diagrams. This chapter is too brief to be of great service to readers. Thus, the whole section on the Spherical Projection could be eliminated. The section on the Stereographic Projection should be greatly expanded to show students and others just how optical data are plotted in a stereographic projection. Reference to the treatment in Johannsen's "Manual of Petrographic Methods," Boeke's "Die Anwendung der stereographischen Projektion bei kristallographischen Untersuchungen" and F. C. Phillips "An Introduction to Crystallography" would have been helpful. The section on Gnomonic Projection could be eliminated.

An error present in earlier editions of this book and in "The Microscopic Characters of Artificial Inorganic Solid Substances or Artificial Minerals" is continued here and appears on page 10. The composition of point P_3 is incorrectly given. It should be 50 per cent B, 30 per cent A, and 20 per cent D. There is no component E in this hypothetical system.

The main part of the book is called the "Description of Minerals" and comprises ten parts; I. Elements; II. Halides; III. Sulfides; IV. Oxides; V. Carbonates, Nitrates, and Iodates; VI. Other mineral Carbon Compounds; VII. Borates; VIII. Sulfates, etc.; IX. Phosphates, etc.; X. Silicates. These major divisions in the classification are further subdivided on the basis of chemical and structural principles. The subjects covered for each substance are given below together with comments.

(1) *Name, Crystal class name, Chemical formula, Crystallographic elements of crystallization*

The crystal class names used are those of Groth modified by Rogers. The authors are to be congratulated on this choice which is almost universal today.

(2) *Composition*

(3) *Structure*

This includes the space group, sides of the unit cell, and the number of formula atoms in the unit cell. The errata sheet contains many corrections to these data. Professor D. J. Fisher, in a review in *Jour. Geol.*, **59**, no. 4, pp. 402–407, 1951, gives a large number of corrections covering the structural data.

(4) *Physical characters*

Crystal habit, twinning, parting, cleavage, hardness, specific gravity, fusibility, solubility, magnetism are given where data are available.

(5) *Optical Properties*

These are given in detail for opaque and non-opaque minerals. The reflection percentages of Schneiderhöhn and Ramdohr and others are given for opaque minerals. The data of R. Bailly on the optical properties in the infra-red have also been added.

(6) *Alteration*

(7) *Occurrence*

This subject receives a very uneven treatment. For some minerals, such as lazulite and scorodite, no localities are given. For others, such as molybdenite, as many as three localities are given. In other references, the given localities are so general as to be of slight value. Thus, borax is stated to occur in "certain salt deposits, as in Tibet and California"; chrysoberyl "found in pegmatite in New England, etc., also in micaceous schists in the Urals, also in alluvial deposits in Brazil or Ceylon"; turquoise, "found in veins in trachyte in Persia, in copper ore deposits, as in Chile." The choice of localities is not always based on the size of the deposit, its fame, nor on the quality of the material for research purposes. This reviewer believes that localities for optically and chemically described material are very helpful for those who are teaching or engaged in research, and should be given in some detail.

(8) *Diagnostic characteristics*

Illustrations

Many new graphs relating optical properties to composition are given in this book. Not all will agree with the basis for the construction of a number of these graphs: but, as the Winchells state on page xiv that some of these graphs must be considered "*rather crude approximations*," it will be up to the reader to evaluate these charts. Nineteen photomicrographs are included, but unfortunately no locality is given with any of these illustrations.

Classification

The classification is strongly influenced by the two leading modern treatises, Dana's "System of Mineralogy," 7th Edition, and Hugo Strunz' "Mineralogische Tabellen."

The chapter on the silicates has an eight page introduction to the classification of these minerals which is based on crystallo-chemical considerations. The classification adopted is that of Hugo Strunz with the addition of M. Fleischer's class of cyclosilicates for minerals having rings of tetrahedrons in their structure. Quartz, tridymite, cristobalite, and opal are classed as tectosilicates.

General Comments

There are a number of matters concerning nomenclature, classification, and interpretation in this book which are totally at variance with the opinions of many mineralogists. Only a few of these differences are presented here:

In the preface on page vi, one reads with surprise the statement "This concept that one mineral may vary in composition *is a new one;* indeed, at the start of this century most mineralogists believed that any one mineral had one fixed composition, and . . . " (the italics are the reviewer's). It apparently needs to be re-emphasized that Yale University was a stronghold, in the United States, in the study of the variation in the physical properties of minerals with a variation in chemical composition. Thus, in the *American Journal of Science* in 1894, Penfield presented a paper "On the crystallization of herderite" in which he relates change of optical properties to substitution of OH for F; this was followed by one entitled "On the chemical composition and related physical properties of topaz." In 1895, Penfield and Pratt discussed "The effect of the mutual replacement of manganese and iron on the optical properties of lithiophyllite and triphyllite. In 1896, Penfield and Forbes wrote on "Fayalite from Rockport, Massachusetts," and on the "Optical properties of the chrysolite-fayalite group and of monticellite." These contributions serve to illustrate the point that the variation in the chemical composition of minerals was well recognized in the 1890's. Ford continued such studies at Yale and Penfield's influence also took a firm hold elsewhere, as at the U. S. Geological Survey and the Geophysical Laboratory.

(2) In speaking of *solid solution* relationships between fluorite, YF_3, and CeF_3, one is led to believe that there is a difference between the synonyms, *crystal solution* and *mix-crystals*. The usage of the synonyms elsewhere in the book is confusing. It would be better to select one term for solid solution and adhere to the usage.

(3) The coverage of the feldspar group, 59 pages, is very detailed. Although reference is made to the work of Dittler and Köhler, and Köhler and Tertsch on the character of high-temperature feldspars, no attempt is made to interpret these papers or to treat the modern developments in this field. Considering the importance of recent investigations, this section is somewhat disappointing.

(4) The statements "The hydration of feldspars may lead to the formation of sericite, kaolinite, zeolites or similar minerals," p. 261, and "By hydration muscovite alters to damourite or hydromica," p. 368, and similar statements in the book are not in keeping with the advances in physical chemistry in the last thirty years. The process of alteration is far more complex than simple hydration.

(5) The treatment of the clay minerals is very disappointing. A large amount of careful work has been done on the minerals of the montmorillonite group. Figure 275 is of little use. The formulas for the montmorillonite group are difficult to interpret since a large number of proposed formulas have been suggested in an effort to include the variable interlayer water as a part of the formula. Thus, the generally accepted formulas of Ross and Hendricks have not been adopted. The usage of the term "leverrierite" is in error. Leverrierite is a synonym of kaolinite. Miloschite has been shown by Grim and Rowland to be a mixture. The clay mineral brammallite is a true hydromica, a sodium-rich variety, and is most certainly not to be classified with paragonite. In the classification of the montmorillonite group on page 398, canbyite is included under nontronite; but on page 400, in the description of canbyite, it is stated to be related to kaolinite. This is very confusing. In footnote 156 on page 399, it is stated that "Saponite and sauconite are included here (although anhydrous) because they are so closely related to montmorillonite." Ross clearly showed from his analyses that these minerals are hydrous and he demonstrated unquestionably that they are members of the montmorillonite group.

There is no treatment of the hydromica group. Hydromica, as such, is mentioned under the alteration of muscovite on page 368. Bravaisite is relegated to a footnote on page 377, while the group name "illite" is not even listed. No mention is made of glimmerton. Damourite, a variety of muscovite, is made the equivalent of hydromica.

(6) The system of nomenclature followed by A. N. Winchell for a series of minerals composed of two end-members will not find general acceptance. The definition is as follows: "The name for a mineral species is a name which applies to all variations in composition which are possible in the given phase; it is a name of a natural unit." Thus the mineral *enstenite* applies to the entire series of orthorhombic pyroxenes from $MgSiO_3$ to $FeSiO_3$, and hypersthene and enstatite are considered as mere varieties or subspecies. Other examples of this are:

The mineral triphyllite includes the variety lithiophyllite
The mineral barytocelestite includes the varieties barite and celestite
The mineral alum includes the varieties potassalumite and tschermigite
The mineral scorodite includes the varieties scorodite and mansfieldite

The mineral variscite includes the varieties variscite and strengite
The mineral triploidite includes the varieties triploidite and wolfeite
The mineral childrenite includes the varieties childrenite and eosphorite

But note that:

(1) Phosphosiderite and metavariscite are given equal status.
(2) Rockbridgeite and frondelite are given equal status.
(3) The members of the vivianite group are broken down into six types.

This attempt, by Professor A. N. Winchell, to be logical in problems of nomenclature has led to some illogical results.

George T. Faust
U. S. Geological Survey, Washington, D. C.

ROCKS AND MINERALS, Vol. 26, Nos. 9–10, September–October, 1951. 25th Anniversary Issue.

A noteworthy effort toward bridging the gap between the interests of professional and amateur mineralogists is the 25th Anniversary Issue of *Rocks and Minerals*, edited by Clifford Frondel and Arthur Montgomery and presented as a tribute to the editor and publisher, Peter Zodac. Contributors include Arthur Montgomery, Henry S. Canby, Charles Palache, Charles R. Toothaker, Richard V. Gaines, the late Lloyd W. Fisher, A. Pabst, C. D. Woodhouse, M. Vonsen, George Switzer, Samuel G. Gordon, Mark C. Bandy, David M. Seaman, Arthur J. Boucot, and Elmer B. Rowley.

Articles of particular readability and interest to the reviewer are those by Gaines, The Sapphire Mines of Kashmir, and by Bandy, The Ribaue—Alto Ligonha Pegmatite District, Portugese East Africa. Both of these articles present new information on what were heretofore largely unknown mineral districts. Most of the articles are well written and both line drawings and plates are far above average for a publication of this general rank. The co-editors deserve the thanks of all mineralogical enthusiasts for their unstinting efforts toward making the Anniversary Issue completely effective and worthwhile.

E. Wm. Heinrich
Univ. of Michigan, Ann Arbor, Mich.

PHASE TRANSFORMATIONS IN SOLIDS, by R. Smoluchowski and others, ix+659 pp., 92 figs., New York, John Wiley & Sons Inc. (1951). $9.50.

This book is most difficult to review. It consists of seventeen unrelated essays on seventeen aspects of phase transformations in solids. All are of high quality, by authors of recognized authority, and each would merit an individual discussion.

Some are purely theoretical, and of these "On the General Theory of Phase Transitions" by Laszlo Tisza appealed to the reviewer as of special interest. More specific are the essays by Kracek, Schairer, and Weyl respectively on one-component silicate systems, two-component silicate systems, and glasses. The several essays dealing with transformations in metals are excellent.

The book can unhesitatingly be recommended.

George W. Morey,
Geophysical Laboratory, Washington, D. C.

NEW MINERAL NAMES

Harkerite

C. E. Tilley, The zoned contact-skarns of the Broadford area, Skye: a study of boron-fluorine metasomatism in dolomites: *Mineralog. Mag.*, **29**, 621–666 (1951).

Chemical: Analysis by H. C. G. Vincent gave SiO_2 14.17, B_2O_3 7.77, Al_2O_3 2.84, Fe_2O_3 0.85, FeO 0.46, MnO 0.02, MgO 11.15, CaO 46.23, CO_2 14.94, Cl 1.36, H_2O^+ 0.81, H_2O^- 0.11; sum 100.71, less $O=Cl$ 0.31, 100.40%. The formula is discussed; a rough fit with the unit cell is given by the following:

$20CaCO_3 \cdot Ca_{28}(Mg, Al, etc.)_{20}(B, Si)_{24}(O, OH, Cl)_{96}$. The Mg group has Mg 15.65, Al 3.16, Fe''' 0.60, Fe'' 0.36, the (B, Si) group has Si 13.35, B 12.63.

The mineral dissolves with effervescence in acetic or hydrochloric acids; gives a good flame test for boron. Heated to 850°, it decomposes to a turbid brown product.

Crystallographic and *X*-Ray Data: *X*-ray study by N. F. M. Henry give the Laue group $m3m$ and the tests for pyro- and piezo-electricity were negative. The crystal class is probably $m3m$ (cubic holohedral). The cell has $a=29.53\pm.01$ Å., with a pseudo-cell at 14.76 Å. This pseudo-cell contains the formula given above. *X*-ray powder data are given: the strongest lines are (1) 2.61, (2) 1.84, (3) 2.13, (4) 1.51 Å.

Physical and Optical: Harkerite occurs typically as simple octahedra that are colorless with vitreous luster, but alter to white masses of calcite. Isotropic, with n 1.653, but some sections show anomalous birefringence, like that of some garnets from contact zones. $G=2.959$. No cleavage.

Occurrence: Occurs in a skarn containing monticellite, calcite, and accessory bornite, chalcocite, magnetite, and diopside, at the contact of dolomitic limestones with Tertiary granite, Isle of Skye.

Name: For Alfred Harker, 1859–1939, British petrologist.

Michael Fleischer

Eckrite

J. Ravier, Sur une nouvelle variete d'amphibole: l'eckrite: *Bull. soc. franc. mineral. et crist.*, **74**, 10–19 (1951).

Amphibolite containing more than 80% amphibole, with quartz, biotite, sphene, apatite, and magnetite, fills a fault in migmatized gneiss in the Eque region on the west coast of Greenland, opposite Disko Island. Analysis of the amphibole (Patureau—analyst) gave SiO_2 54.40, Al_2O_3 0.80, Fe_2O_3 11.80, FeO 4.35, MgO 16.00, CaO 6.20, Na_2O 3.45, K_2O 0.95, MnO 0.16, TiO_2 0.80, P_2O_5 0.22, H_2O^- (100°) 0.15, H_2O^+ 1.70; sum 100.98%. This corresponds to $NaCaFe'''(Mg_{3.5}Fe''_{0.5})(Si_4O_{11})_2(OH)_2$. The mineral has n approx. 1.6, birefringence about 0.02, strong pleochroism with X violet-blue, Y sky-blue, Z pale yellow, 2V neg., variable 0 to 35°, $X{:}c$ about 40°. These properties are compared with those of amphiboles such as glaucophane, arfvedsonite, riebeckite, etc., with the conclusion that none is exactly like this one. The name is for the Eque region.

Discussion: Additional variety names such as this are not justified unless a thorough review of the whole group is made and the variety names are considered systematically.

M. F.

Magniotriplite

A. I. Ginsburg, N. A. Kruglova, and V. A. Moleva, Magniotriplite—a new mineral of the triplite group: *Doklady Akad. Nauk S.S.S.R.*, **77**, 97–100 (1951); from a translation kindly made by Mr. V. L. Skitsky.

The mineral occurs as crystals and as masses up to 0.5×8 cm. in pegmatites of the

Turkestan Ridge. It is reddish-brown, luster vitreous, one cleavage observed under the microscope, fracture uneven, hardness 4, G.=3.57. Analysis FeO 25.90, MnO 13.00, MgO 17.12, CaO none, Al_2O_3 0.70, alkalies none, P_2O_5 36.52, SiO_2 (quartz) 2.40, TiO_2 0.92, H_2O^+ 0.64, H_2O^- 0.10, F 6.00; sum 103.30 minus O=2F 2.52, 100.78%. This leads to the formula $(Mg, Fe, Mn)_{15}(PO_4)_8F_6$ with Mg:Fe:Mn=0.425:0.360:0.183. (The usual formula is $R_2(PO_4)F$). Optically the mineral has $nX=1.641$, $nY=1.649$, $nZ=1.661$, $X:a=18°$, $Z=b$, pleochroic with X=light yellow, nearly colorless, Y=light yellow, Z=wine-yellow, 2V 60°, pos.

Discussion: This is outside the range of composition plotted for triplite by Heinrich, *Am. Mineral.*, **36**, 256–271 (1951). Previous analyses of triplite with high MgO have all been high in Fe and low in Mn.

M. F.

Magnalumoxide

N. A. Bobkov and Yu. V. Kazitsyn, The new mineral magnalumoxide: *Vses. Mineral. Obshch. Zapiski* (*Mém. soc. russe minéral.*) **80**, 108–121 (1951); from a translation kindly made by Mr. V. Skitsky.

The mineral occurs in octahedral crystals up to 15 cm. in diameter, showing the forms {111}, {110}, and {113} only. The crystals are black with vitreous luster, streak gray-green. Sp. gr. 3.76, hardness 7½–8. Optically isotropic with n 1.745. *X*-ray powder photographs gave a 8.086±.002Å; some lines are not accounted for by this cell size and a superstructure may be present. Analysis gave SiO_2 0.75, Al_2O_3 68.56, Fe_2O_3 11.37, Cr_2O_3 0.05, FeO 0.99, MgO 15.55, MnO 0.41, CaO 0.93, TiO_2 0.30, P_2O_5 0.02, Na_2O 1.34; sum 100.27%. This corresponds to 53.3 mol % $Mg(Al, Fe)_2O_4$, 46.7 mol % $(Al, Fe)_2O_3$, or nearly $Mg_5(Al, Fe)_{18}O_{32}$. Solid solutions of alumina (as gamma-alumina?) in spinel were long ago reported in synthetic studies, but had not previously been reported as naturally occurring minerals. The crystal structure was investigated in great detail; it differs from the spinel structure and the space group is probably T_d^1. The mineral occurs in a metasomatic vein which also contains diopside, phlogopite, and calcite, and which cuts a marble-diopside rock in the area of the Gon River, within the Dzheltulinsk metamorphic section of the Aldan shield, southeastern Siberia.

The name is from the composition.

M. F.

Taaffeite

B. W. Anderson and G. F. Claringbull, Taaffeite, a new beryllium mineral, found as a cut gem-stone: *Mineralog. Mag.*, **29**, 765–772 (1951).

A small (1.419 carats) mauve gem-stone, supposedly spinel, was noted by Taaffe to show double refraction. Microchemical analysis by M. H. Hey gave Al_2O_3 70.0, Fe_2O_3 5.9, MgO 13.4, BeO 11.0, corresponding to $BeMgAl_4O_8$, intermediate between spinel and chrysoberyl. *X*-ray powder data are given; the strongest lines are 2.43, 2.05, 1.05 Å. Laue and oscillation photographs gave space group $D_6^6-C6_32$, $a=5.72$, $c=18.38$ A, c/a 3.21. Sp. gr.=3.613±.002. The unit cell contains $Be_4Mg_4Al_{16}O_{32}$. The mineral is optically negative, with nE 1.7182, nO 1.7230; a second sample had nE 1.7167, nO 1.7208. Measurements of dispersion gave for nO:

λ	6708	5893	4226 Å
nO	1.7191	1.7230	1.7390

(The locality is unknown, but the shape suggested that the stone had been cut in Ceylon.)

The name is for Count Taaffe, "a brilliant if unorthodox Dublin gemmologist," who first noted the unusual properties of the stone.

M. F.

Andersonite

J. M. Axelrod, F. S. Grimaldi, Charles Milton and K. J. Murata, *Am. Mineral.*, **36**, 1–22 (1951).

Bayleyite

J. M. Axelrod et al., *Am. Mineral.*, **36**, 1–22 (1951).

Swartzite

J. M. Axelrod et al., *Am. Mineral.*, **36**, 1–22 (1951).

Huttonite

Adolf Pabst and C. O. Hutton, *Am. Mineral.*, **36**, 60–69 (1951).

Sabugalite

Clifford Frondel, *Am. Mineral.*, **36**, 671–679 (1951).

Novacekite

Clifford Frondel, *Am. Mineral.*, **36**, 680–686 (1951).

Ferrocarpholite

W. P. de Roever, *Am. Mineral.*, **36**, 736–745 (1951).

M. F.

The following new names are used in *Dana's System of Mineralogy,* 7th Edition, Vol. 2 (1951).

Bararite (p. 106)

Name given to the hexagonal dimorph of $(NH_4)_2SiF_6$, formerly included with the isometric modification under the name cryptohalite. The name is for the locality, Barari, India.

Cobaltocalcite (p. 175)

Name given to $CoCO_3$, formerly called sphaerocobaltite. The latter is considered unsatisfactory "because of the suggested relation to cobaltite and the use of the mode of aggregation as a species rather than as a varietal designation."

Chalcocyanite (p. 429)

Name given to anhydrous $CuSO_4$, formerly called hydrocyanite. Named for the Greek for copper and azure-blue. "The original name is objectionable for an anhydrous substance."

Pentahydrite (p. 492)

Name given to $MgSO_4 \cdot 5H_2O$, not previously named (see *Am. Mineral.*, **36**, 641 (1951)). Cuprian varieties are known.

Davisonite (p. 939)

Name given to $Ca_3Al(PO_4)_2(OH)_3 \cdot H_2O$ described by Larsen and Shannon, *Am. Mineral.*, **15**, 322 (1930), as dennisonite. Named for J. M. Davison (1840–1915) of the University of Rochester whose name was erroneously written as Dennison.

Meta-uranopilite (p. 582)

Name given to $(UO_2)_6(SO_4)(OH)_{10} \cdot 5H_2O$, formerly called beta-uranopilite. Presumably changed because it is less hydrated than uranopilite, not a dimorph of it.

Metastrengite (p. 769)

Name given to $FePO_4 \cdot 2H_2O$, called phosphosiderite since 1890. Name changed to parallel the nomenclature variscite-metavariscite.

Hydroxyl-herderite (p. 820)

Name used to replace hydro-herderite.

Changes in Hitherto Usually Accepted Nomenclature in *Dana's System of Mineralogy, 7th Edition, Vol. 2*

Natromontebrasite (in place of Fremontite) (p. 823)
Ammonia Alum (in place of the Tschermigite) (p. 475)
Devillite (in place of Herrengrundite) (p. 590). See discussion, *Am. Mineral.*, **26**, 293 (1941)

M. F.

DISCREDITED MINERALS

Nuevite (=Samarskite)

Joseph Murdoch, *Am. Mineral.*, **36**, 358–359 (1951).

Stuetzite (=Empressite)

R. M. Thompson, M. A. Peacock, J. F. Rowland, and L. G. Berry, *Am. Mineral.*, **36**, 458–470 (1951).

Erinite (=Cornwallite)

L. G. Berry, *Am. Mineral.*, **36**, 484–503 (1951).

Leucochalcite (=Olivenite)

L. G. Berry, *Am. Mineral.*, **36**, 484–503 (1951).

Stylotypite (a mixture, mainly tetrahedrite)

Charles Milton and J. M. Axelrod, *Am. Mineral.*, **36**, 696–703 (1951).

M. F.

NEW DATA

Jordisite (Amorphous molybdenum sulfide)

L. W. Staples, *Am. Mineral.*, **36**, 609–614 (1951).

M. F.